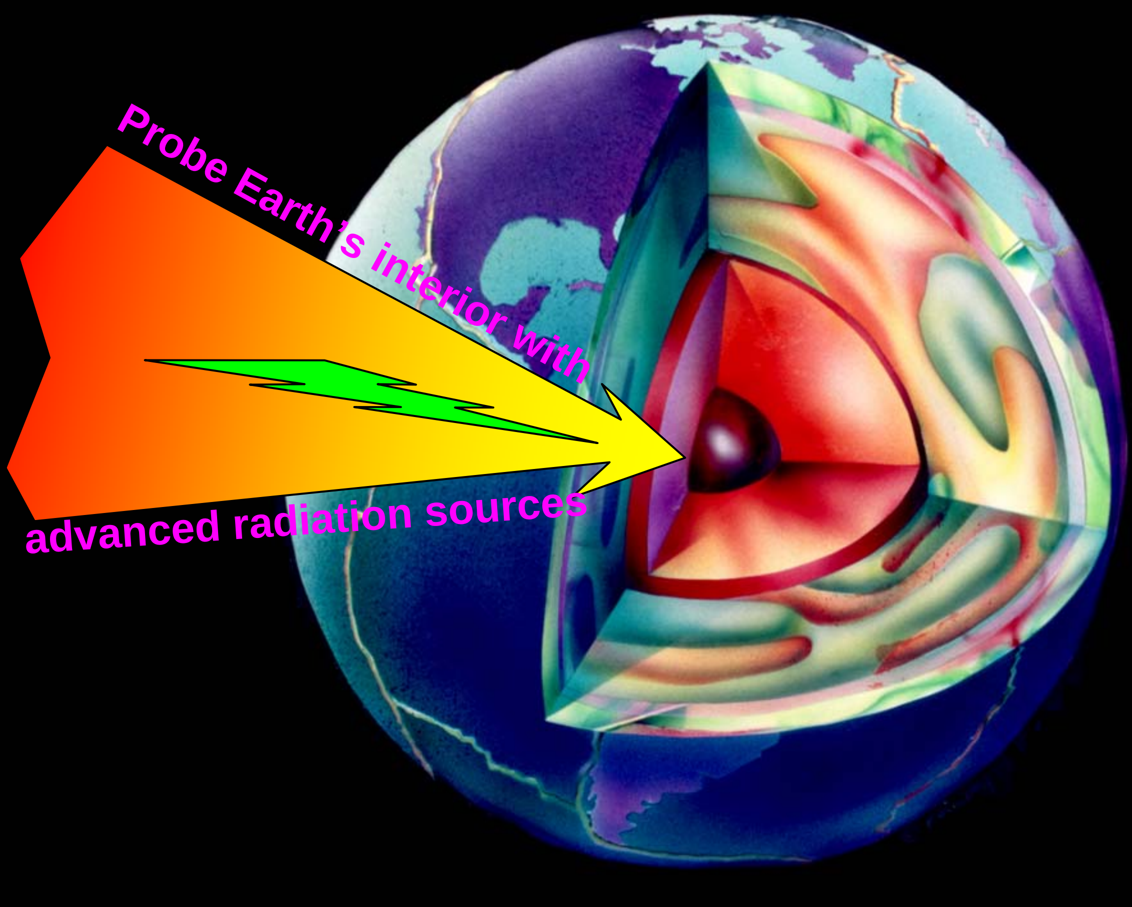
COMPRES

Community Facilities and Infrastructure Development for High-Pressure Mineral Physics and Geosciences: COMPRES Ⅱ



Proposal to NSF 2007-2012

Part A: Project Overview

Part B: Program Reports

About the Cover

The cover for this proposal was designed by Jiuhua Chen, with contributions from Michael Vaughan. The Earth cutaway diagram was created by Keelin Murphy for education and outreach programs of the Mineral Physics Institute at Stony Brook University and has been widely disseminated throughout the world.

The composite image illustrates the fundamental approach of members of the COMPRES community of mineral physicists: we use experimental facilities installed at the advanced radiation sources [synchrotron X-rays and neutron radiation] at the national laboratories of the Department of Energy to probe the Earth and further our understanding of its physical properties and dynamical processes.

About this Proposal

This proposal was produced by the COMPRES Executive Committee and COMPRES staff members on behalf of the 50 U. S. member institutions of the Consortium. The proposal consists of three parts: A. Project Overview; B. Program Reports; and C. Research Accomplishments. It has been developed over the past 14 months under planning procedures established by the Electorate in June 2005.

Part A: Project Overview

Part A serves as a guide to the overall proposal. It is heavily annotated with references to the location(s) of more detailed information provided elsewhere in the proposal. We hope this facilitates reviewer access to the details of primary interest to each reader. In particular, Part A includes a summary of scientific highlights and technological advances in the first 4 years of COMPRES and the challenges for the next 5 years, an overview of the COMPRES Consortium, information on the member institutions and new faculty in mineral physics, management and organization, meetings and workshops, education and outreach activities, information technology and communications, and a list of publications.

This part concludes with a presentation of the Program Plan and budget request for the next five years. The Budget Plan includes our estimates of the costs to carry out the activities that are summarized in the Project Overview and detailed in the Program Reports.

Part B: Program Reports

Part B consists of detailed progress reports on the Community Facilities operations and Infrastructure Development projects supported by COMPRES in the period 2002-2007 and proposed plans and detailed budgets for the period 2007-2012.

Part C: Research Accomplishments

Part C is a compilation of one-page summaries (One-Pagers) which report highlights of the scientific and technological achievements of students, staff and faculty researchers using COMPRES-supported facilities, data produced from COMPRES programs, and the infrastructure development projects supported by COMPRES in its first four years from 2002-2006.

This proposal was prepared by Ann Lattimore, Administrative Coordinator for COMPRES, with assistance from Samantha Lin and Michael Vaughan. The design and formatting of Part C was done by Glenn Richard.

Community Facilities and Infrastructure Development for High-Pressure Mineral Physics and Geosciences: COMPRES II

May 1, 2007—April 30, 2012

Submitted to
National Science Foundation
Division of Earth Sciences
Instrumentation and Facilities Program

By
Consortium for Materials Properties
Research in Earth Sciences
ESS Building-Room 167
Stony Brook, NY 11794-2100

On behalf of the
Executive Committee
and 50 Member Institutions
of the COMPRES Consortium

August 2006

ACRONYMS USED IN COMPRES II PROPOSAL TO THE NSF—AUGUST 2006

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 ALS
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 under 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
XRD	X-Ray Diffraction

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Supplementary Documents for submission via FastLane:

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2. **Poster based on Bass Report (April 2005)**
3. **EOS article (October 2005): "The Future of High-Pressure Mineral Physics"**
4. **Publications List—as referred to in Section A. 9 above.**
5. **Report of High Pressure Summit Meeting (November 2005)**
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Part A: Project Overview

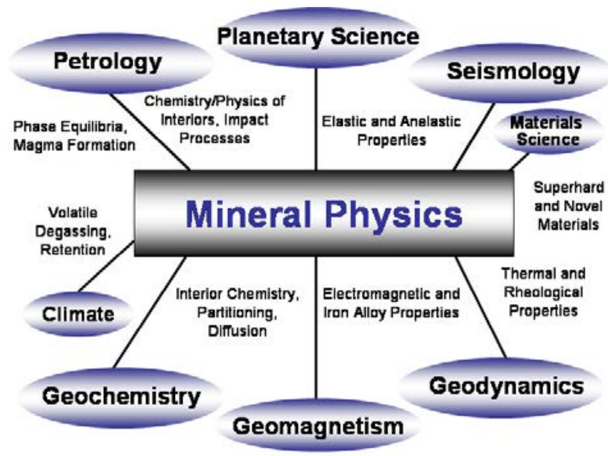
A.1 Introduction and Background

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.

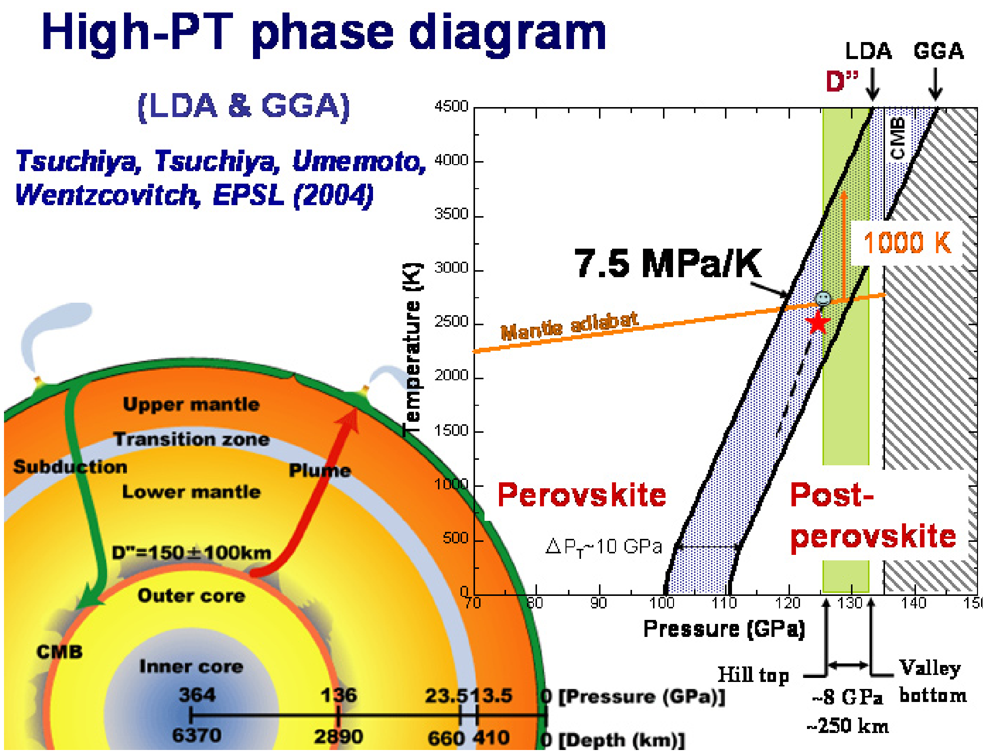


Links of mineral physics with other Earth science fields (EOS, October 4, 2005)

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



High pressure-temperature phase diagram for $MgSiO_3$, with transition from perovskite to post-perovskite phase occurring near 126 GPa, just above the top of the D” layer near the core-mantle boundary (Tsuchiya, Tsuchiya, Umemoto, and Wentzcovitch, Earth Planet. Sci. Lett., 224, 241, 2004]

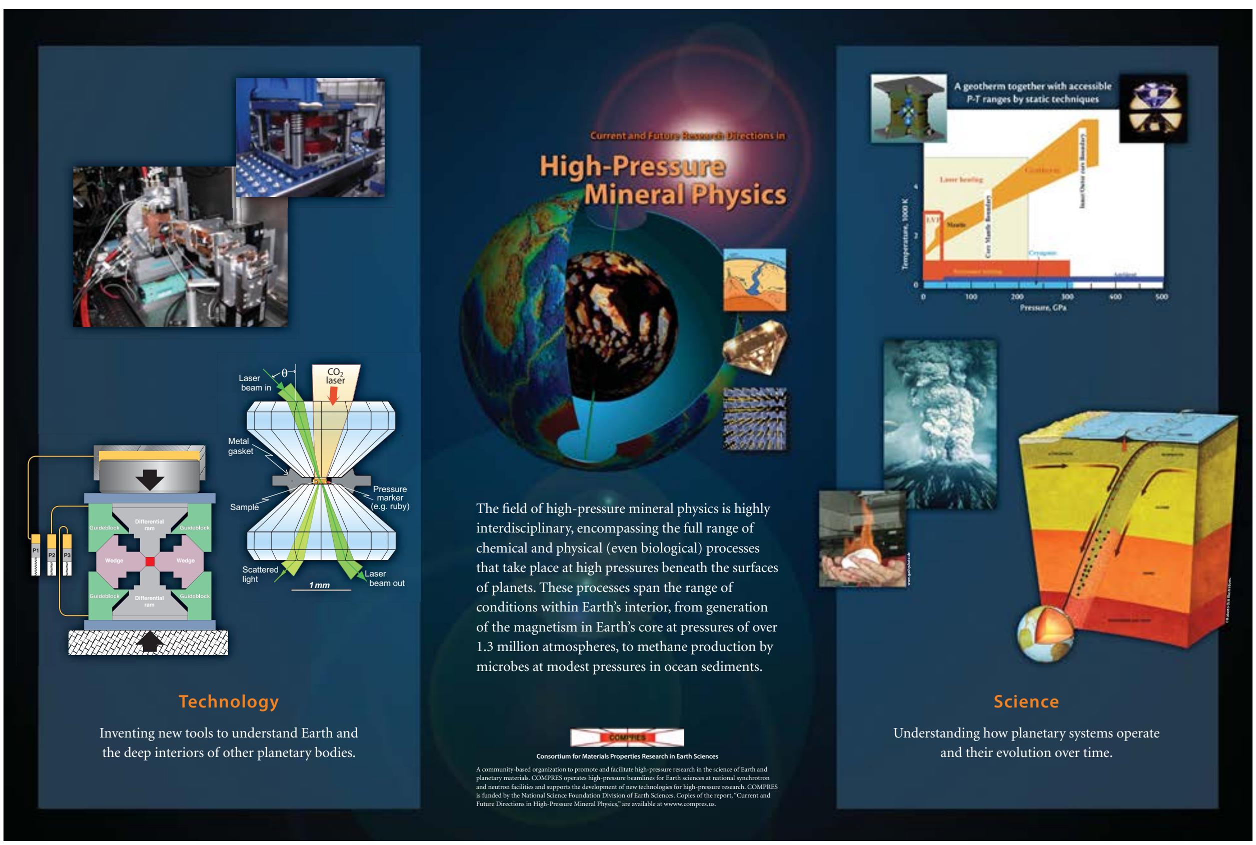
community; the US now competes on equal footing with the Japanese and Europeans who had a considerable jump on this country at the time of the birth of COMPRES. Indeed, the successes flowing from the rapid growth of COMPRES are already feeding back into the international community, as documented in A.3 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 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.



Poster based on Bass Report illustrating "Current and Future Research Directions in High-Pressure Mineral Physics."

A.2 Research Themes, Scientific and Technological Advances, and Challenges for the Next Five Years

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

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.

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 in 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 [2002-2006], 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 $CaCl_2$-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 SiO_2 pressure up to 60 GPa, which provided direct experimental evidence for softening of the elastic shear modulus. [B.1.b]

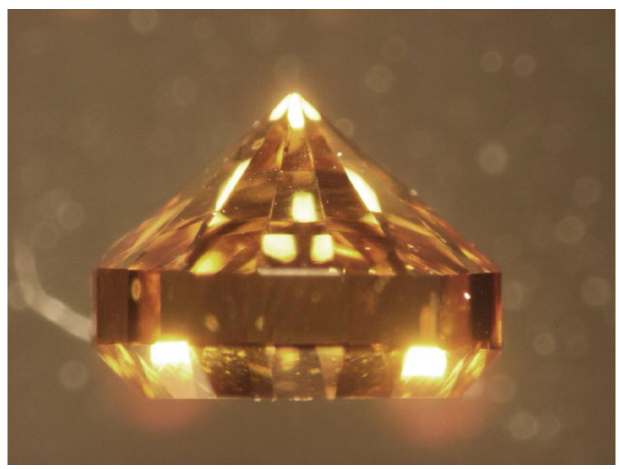
• A. Goncharev and colleagues measured optical spectra of single crystals of (Mg,FeO) to pressures exceeding 60 GPa. They observed enhanced absorption in the mid- and near infrared spectral range, effectively blocking much of the light compared with low pressure conditions, which appears to require high 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 than have been previously inferred. [B.2.d]

• Construction of a completely a 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, Zha and Hemley, *Phys. Stat. Solidi (a)* 201, R25, 2004]. See details in October 2004 Newsletter of COMPRES. [B.1.b]

Abby Kavner and Nathalie Conil from UCLA at beamline 12.2.2 of the ALS.



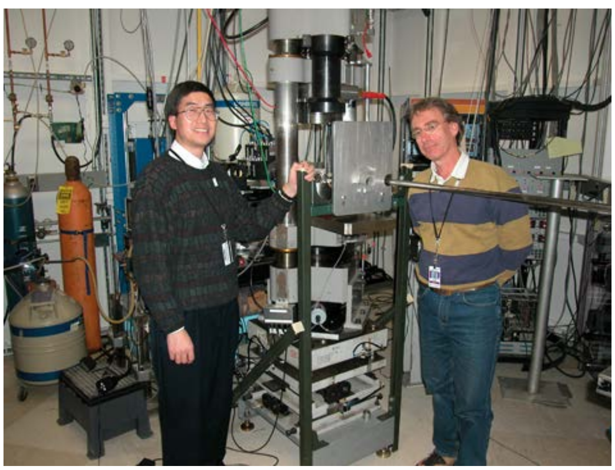
Single-crystal diamond grown by chemical vapor deposition at the Carnegie Institution of Washington.

• 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.2.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].



Shenghua Mei and Bill Durham with D-DIA apparatus installed on beamline X17B2 at the NSLS.

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.l].

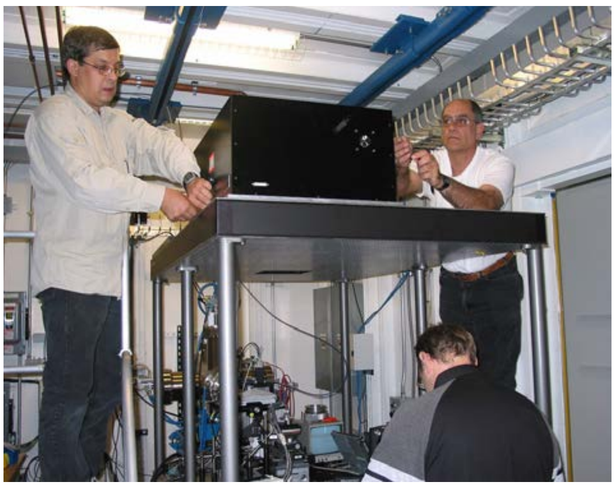
• Ultrasonic interferometry measurements of elastic wave velocities in conjunction with synchrotron X-radiation have been pioneered by B. Li and colleagues in a DIA-cubic anvil apparatus on the X17B2 beamline to simultaneous pressures of 10+ GPa and temperatures above 1000K. These techniques have now 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].

Machinist Brian Nagy and Kurt Leinenweber with mini-lathe at ASU.

• A team led by H-k Mao used large gem crystals moissanite (silicon carbide) as anvils in a panoramic cell with >1mm^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]



Stas Sinogeikin, Jay Bass and Vitali Prakapenka installing Brillouin spectrometer on beamline 13-BM-D at the APS.

• 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.

c. *Challenges for the next five years*

Funding challenges

As documented in this proposal, the first era of COMPRES from 2002-2006 has seen rapid growth and expansion as the community has self-identified and become more fully engaged in the experimental operations at the high-pressure facilities at the national laboratories and in the development of new infrastructure [see details in Parts B.1 and B.2 below].

As a consequence of the technological progress at the COMPRES-supported beamlines and increased user demand for beamtime, the requirements for providing funding for these facilities has increased, both for operational support and necessary equipment upgrades.

At the same time, while some of the Infrastructure Development projects have reached completion, there are two of these that merit continuation in COMPRES II: (1) the multi-anvil cell development project at the Arizona State University; and (2) the inelastic X-ray scattering project at the Advanced Photon Source. As testimony to the success of the ASU infrastructure project in COMPRES I, see letter for User Community at end of section B.2.b.

We are also proposing two important new infrastructure initiatives: (1) establish a Commission [or Working Group] on High Pressure to coordinate research and development of new and more precise calibration standards for high-pressure experiments; (2) establish a High Pressure Synergetic Center (HPSynC) at the Advanced Photon Source. HPSynC would operates on a novel infrastructure concept that connects the large national facility and the high-pressure community and is the major new initiative proposed in COMPRES II [see details in A.10 below].

The desire to sustain and grow the capabilities of the beamline operations at the national laboratories and support the existing ongoing and new infrastructure initiatives is reflected in the funding requested for 2007-2012 in A.10 below, from $10.6 million in the past five years to $17.9 million in the upcoming five years.

From the outset of COMPRES in early 2001, it was recognized that research in theoretical and computational mineral physics is of fundamental importance to the field and to the mission of COMPRES on behalf of the community. Computational mineral physics is currently underrepresented in the COMPRES portfolio. We plan to encourage leading practitioner in this field to convene a workshop to identify the most urgent equipment and infrastructure needs for this field. Based on the outcome of the workshop, we hope that together we can evolve a strategy to seek funding for this initiative via a variety of channels. [see additional details in A.10 below].

Science Challenges

The opportunities and challenges for the COMPRES community in the next five years [2007-2012] are continuing to expand, both in terms of refined knowledge emanating from new observational data produced by programs such as EarthScope and from new discoveries within our solar system and beyond. As an update to the section of the Bass report on planetary processes (Supplementary Document #1), we emphasize this new dimension in the following paragraphs.

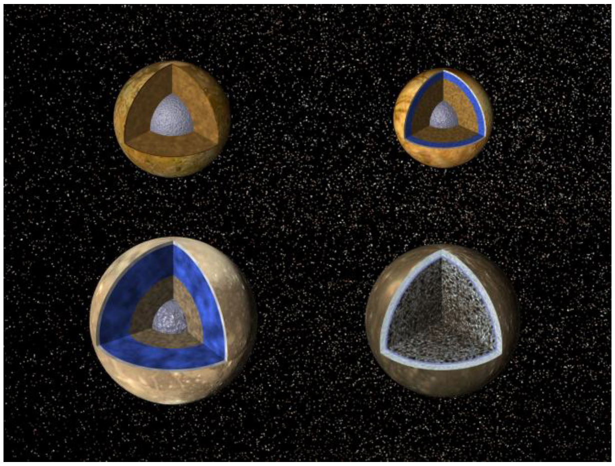
(1) Planetary Science

(a). Properties of Extra-Solar Planets: An Emergent Opportunity

The field of planetary science is undergoing an almost unprecedented paradigm-shift: one that has profound importance both for our understanding of the panoply of possible planetary interiors, and for the future role of COMPRES in facilitating studies of planetary interiors. This shift has been driven by the identification of many dozens of stars with accompanying planets. With the number of known planets increasing by over ten-fold in the past decade, our view of planets has been forced to transition from a model of solar systems driven by our own that involves a Kantian ordered view of concentric, nearly circularly, revolving, interior terrestrial planets, followed by the external gas giant planets to a view involving a cacophony of possible configurations. Using detection techniques that vary from gravitational microlensing to radial velocity measurements of stars using Doppler shifts to observations of stellar intensity variations induced by transiting planets, over 150 extra-solar planets have now been observed (*e.g.* Marcy *et al.*, 2005). These planets encompass not only a range of sizes and orbits, including larger-than-Jupiter sized objects (apparently gas giants) with orbits closer than that of Mercury to their host star, to apparent "super-Earths," likely rocky planets with masses 5.5 and 13 times that of Earth (Beaulieu *et al.*, 2006; Boss, 2006).

This suite of new planets (the sampling of which is undoubtedly strongly biased towards larger planets) poses not only novel high-pressure issues with respect to their internal structure, but also interesting chemical issues as well. The principal controlling factor for whether or not stars are likely to have associated planets are their metallicity: in short, stars with higher abundances of heavier elements are more likely to have detectable planets around them (Marcy *et al.*, 2005). Different stars are expected to have differing chemistries (and thus different planetary chemistries), but the dramatic degree to which they might differ from our own solar system has only recently been appreciated. For example, carbon might be dramatically more abundant than oxygen. A recent study of the apparent composition of the gaseous disk surrounding beta-Pictoris indicates that the C/O ratio is about 16 times the solar ratio (Roberge *et al.*, 2006). By implication, there may be extra-solar terrestrial-like planets whose interiors could be dominated by carbides, rather than oxides.

The opportunities and challenges posed for COMPRES by these astronomical developments are extraordinary. Tantalizing hints exist from both theory and shock experiments at extreme conditions that new phase transitions, and perhaps even new chemical species, may occur in oxides at conditions exceeding those in Earth's interior (Umemoto *et al.*, 2006; Hicks *et al.*, 2006). The key question that emerges is: what phases, with what equations of state, are likely to be present within extra-solar planets? From an experimental standpoint, this question can be subdivided into two parts—(1) What are the properties of hydrogen and helium at the extreme conditions of giant planet interiors (*e.g.*, Gregoryanz *et al.*, 2003); and (2) What are the properties of candidate materials such as oxides, carbides, iron-rich metal, 'ices' (ammonia, water and methane) and their reaction products at pressures and temperatures of, and exceeding those of, Earth's interior? The former question is obviously relevant to planets with masses of approximately Saturn-sized and larger, while the latter is focused on super-Earths, Uranus and Neptune-type planets, and potentially planets of types that have not yet been observed, such as water-rich super-Ganymedes. The currently conceivable pressure- and temperature-regimes can loosely be viewed as 0.1 – 10 Mbar, and thousands of Kelvin—conditions that require the high-intensity x-ray sources and laser heating technologies on which the COMPRES consortium has concentrated. In short, while the maximum reported static pressure is near 5.5 Mbar (Xu *et al.*, 1986), the prospect exists for a large increase in the number of studies at high temperatures in the Megabar and higher range: a development already presaged by the intensive study of the post-perovskite phase. It is studies at these extreme conditions that we expect will be a major developing portion of the second five years of COMPRES.



NASA photo PIA01082

(b). Improving the Understanding of Planets and Satellites in this Solar System

Our understanding of the gas giants will obviously be enhanced by improved high pressure and high temperature equation of state and phase equilibria measurements on hydrogen, helium, and their mixtures described in the previous section. But, separate families of experiments at more modest pressures and temperatures can yield insights into the larger satellites of the gas giants, such as Titan, Ganymede, and Callisto (*e.g.* Scott *et al.*, 2002)—bodies roughly the size of Mercury. In particular, the behavior of fluids, ices and clathrates, and their reactions with coexisting rocky material are of key importance in the chemical evolution of these bodies. By contrast with their giant companions, the typical pressure and temperature ranges within these bodies are fairly modest—less than 8 GPa, with maximum internal temperatures of perhaps 1300 K. Thus, the in situ photon-mediated probes (x-rays and Brillouin and infrared spectroscopies) emphasized by COMPRES have the prospects of yielding a wide suite of new insights into the chemical and physical characteristics of the interiors of these bodies. Key issues that could be resolved by such experiments include: (1) the nature of the fluid that gives rise to the magnetic field of Ganymede; (2) the possible processes that give rise to storage and release of methane within Titan; (3) why Callisto appears to be at least partially undifferentiated; (4) the degree of hydration of the silicate portion of the interiors of the large icy satellites; and (5) the possible composition (and state) of iron-rich cores within these bodies.

Perhaps the most crucial aspect governing how the observed external features of both terrestrial planets and icy satellites correspond to their interiors lies in the rheology of their constituents: again, an area that has been among the principal emphases of the first five years of COMPRES. The possible differences in tectonic style between Venus and the Earth have long been attributed to the effect of water on silicate rheology (Mackwell *et al.*, 1998), and we anticipate that in the second stage of COMPRES, substantially more comprehensive data sets will be brought to bear on the critical role of multi-phase mixtures, trace hydration, grain size, and different flow mechanisms in determining how mantle material within the different terrestrial planets convects. In short, not only will our rheological data be more extensive and of much higher quality, but we also expect a dramatic improvement in its pressure range of the data: COMPRES investigators are at the forefront of extending rheologic measurements to pressures of Earth's deep transition zone (Xu *et al.*, 2003) or, phrased another way, to almost the entire depth extent of the Martian mantle. By the same token, the rheology of ice and particularly ice-clathrate mixtures is likely critical in generating the viscous flow whose surface manifestations are the spectacularly complex terranes present on Ganymede and Titan. The precise flow mechanism of even pure ice remains a topic of active study (Kubo *et al.*, 2006), and that of multi-phase mixtures is truly in its infancy. Again, COMPRES has played, and is poised to play a critical role in facilitating studies of in situ deformation and texture evolution at high pressures within ice and ice-clathrate mixtures.

An improved coupling of elasticity studies with phase equilibria studies of iron alloys, one of COMPRES' traditionally active areas (*e.g.*, Lin *et al.*, 2004), holds the prospect of providing constraints on core compositions of different terrestrial planets. The key recognition that the Martian core is (at least partially) molten, based on an analysis of Mars' Love numbers (Yoder *et al.*, 2003), combined with the long-standing observation of a present-day internally generated magnetic field on Mercury implies that liquid central cores appear to be the rule, rather than the exception, for the terrestrial planets. However, the chemistry (or, more specifically, the possible lighter alloying components) of iron-rich molten cores depends on the differentiation and accretion history of the planet. But, because of the suite of temperatures and pressures at which COMPRES-sponsored facilities are able to monitor the degree of equilibration between iron-rich material and coexisting silicates, there is the prospect of deriving meaningful constraints on the identity of the lighter alloying components of the cores of the different terrestrial planets. Not only would such constraints provide potentially verifiable (or falsifiable) predictions for the densities and thus sizes of planetary cores, but they would provide a basis for accurately assessing what the bulk compositions of our planetary neighbors are: core compositions provide a major uncertainty when inferring the bulk composition of any planet (including Earth).

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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 27 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 [see joint statement of relationship between GSECARS and COMPRES is given in the Supplementary Documents].

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 CO_2 laser-heated diamond-anvil cell; (6) COMPRES en-

US map with Community Facilities and Infrastructure Development Projects sites.

vironment 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 and other annual meetings clearly demonstrate 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

• 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 CO_2 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. A copy of this joint statement is given in the Supplemental Documents for this proposal.

Site visit of COMPRES Facilities Committee to GSECARS at the APS.

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. A copy is included Supplementary Documents.

COMPRES linkages with international programs

The membership of non-US institutions as foreign affiliates of COMPRES has grown from 0 in 2002 to 27 in 2006, 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 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, as reflected in the user lists included in Supplementary Documents.

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].

Set of journal covers of special issues of scientific journals, national reports and monographs

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

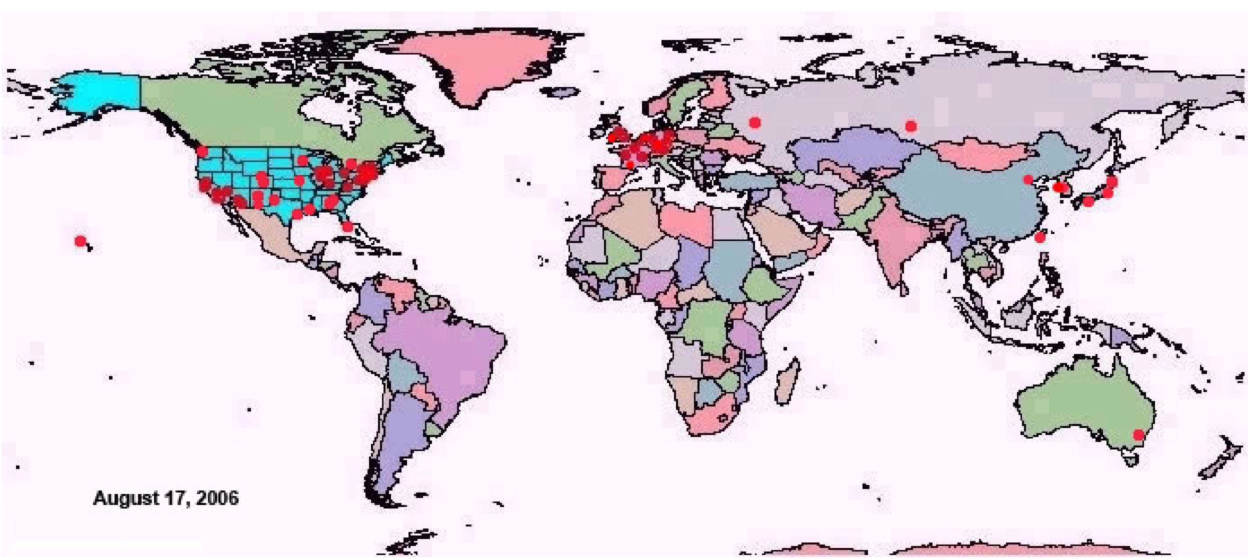
a. Member Institutions

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 27 foreign affiliate institutions. These institutions are listed in a table in the Supplementary Documents for this proposal and indicated on the attached maps.

U. S. map of member institutions of COMPRES



World map of U. S. member institutions and foreign affiliates of COMPRES

b. 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-Mukhapadhyay	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 Geoinstitüt [Germany]
T. Tsuchiya	2006	Ehime University [Japan]
Michael Walter	2004	University of Bristol [United Kingdom]
Wim van Westrenen	2002	Vrije Universiteit [Netherlands]

c. 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.

Details of the work of Jackson, Mao and Li in the One-Pagers in Part C of this proposal. 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

a. 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.

b. 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 July 2006, there are 50 U. S. members; each institution has one vote in any business decisions of COMPRES. There are also 27 foreign affiliate members; these do not have voting rights.

c. 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 Lesher, 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 Polices 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]

e. Central Office

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

The Central office staff includes Robert Liebermann, the President of COMPRES (from September 1, 2003) and Ann Lattimore, Administrative Assistant, both of whom are supported by the COMPRES 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: Jiuhua Chen, Research Associate Professor. COMPRES role: Editor of Newsletter Glenn Richard, Educational Coordinator: COMPRES role: Web Manager and Education/Outreach activities. Michael Vaughan, Research Associate Professor: COMPRES role: Manager of 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

Group photo of the 109 attendees at 2005 Annual COMPRES Meeting at Mohonk Mountain House in New Paltz, NY.

Collage of activities from Granlibakken, Mohonk and Snowbird meetings.

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 Sournce. 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 Presssure 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

Rheology Grand Challenge Workshop

Stony Brook University, August 14, 2006, COMPRES funding $0K

Rachel Dwarski from the University of New Mexico examining a run product at the Multi-Anvil Workshop in March 2005.

Attendees at the Workshop on Synergy of 21st High-Pressure Science and Technology in April 2006

A.7 Education and Outreach

a. 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.

b. Proposed COMPRES REU Summer Scholars 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 selected for this program.

Photo of undergraduate students in REU Summer Scholars program of Mineral Physics Institute of Stony Brook Univesity

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 Summer Scholar 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 Summer Scholar 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.

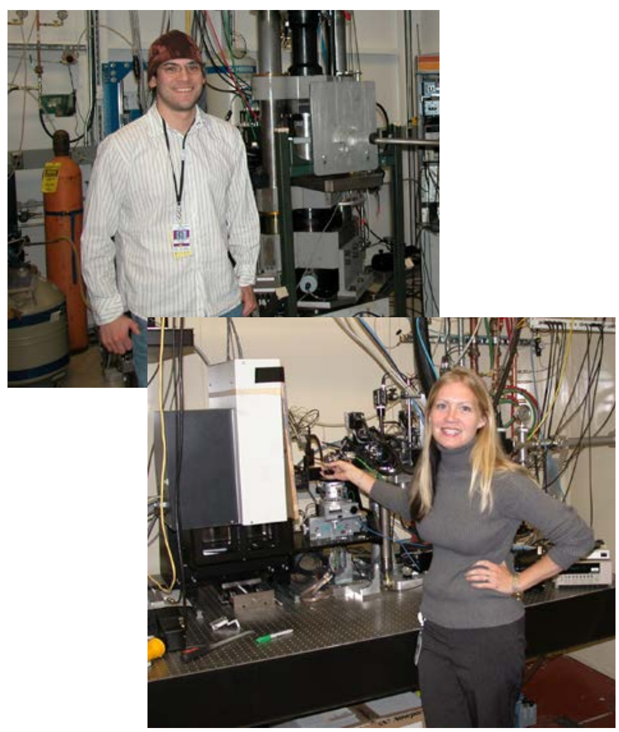
c. Student Interns at Beamlines of National Facilities

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: Jiuhua Chen and Michael Vaughan.
Topic: Density of Molten FeS
Both of these beamline 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.



Student interns Christopher Young at the the X17B2 beamline at the NSLS and Arianna Gleason at the 12.2.2 beamline at the ALS

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.

a. 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 Jiuhua Chen and may be found on the COMPRES web site at www.compres.us/Newsletter/

b. 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.

c. 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. Jiuhua 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 Jiuhua Chen can be found at: www.compres.us/Meetings/2005-12-12-AGU-Powerpoint/COMPRESbooth05.ppt

Collage of COMPRES newsletters

A.9 Publications

In the first 4+ years of COMPRES [2002-2006], 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 Budget Request

This program plan and budget request was developed by the COMPRES community over the past 14 months following procedures and planning timelines established by the Electorate at the 2005 Annual Meeting in Mohonk in June 2005 [see details at the end of this section].

a. Program Plan for 2007-2012

In the five-year period of COMPRES II [2007-2012], we propose to continue the three-fold structure and programs of the Consortium:

(1) Community Facilities Operations

(2) Infrastructure Development Projects

(3) Community Activities
plus administrative office operations.

(1) Community Facilities Operations

COMPRES proposes to continue to support the operations of high-pressure beamlines at synchrotrons to provide access and support to faculty, students and staff scientists in the US Earth Science community. These operations include: (a) Diamond-anvil X-ray facilities at the Advanced Light Source [ALS] of the Lawrence Berkeley National Laboratory; (b) Diamond-anvil X-ray and (c) Infrared facilities at the National Synchrotron Light Source [NSLS] of the Brookhaven National Laboratory; (d) Multi-anvil X-ray facilities at the NSLS; (e) COMPRES also proposes to continue 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. These operations are coordinated with those of the GeoSoilEnviroCARS [GSECARS] program of the University of Chicago at the Advanced Photon Source of the Argonne National Laboratory. We also anticipate that, as was the case during COMPRES I, certain COMPRES-funded infrastructure may be placed at GSECARS if the community believes that is the best national location for a particular new instrument.

In Part B.1 below, we present detailed progress reports on the Community Facilities operations at the national laboratories supported by COMPRES in the period 2002-2007 and proposed plans and detailed budgets for the period 2007-2012.

(2) Infrastructure Development Projects

In addition to the operation of community facilities, COMPRES proposes to provide support for infrastructure projects to promote the development of new technologies for high-presssure research, for use both in laboratories of our home institutions and at the national laboratories. Current examples of such infrastructure development projects include: (a) Pressure calibration at high temperature; (b) Multi-anvil cell assembly development; (c) Brillouin spectrometer for the APS; (d) Nuclear resonant inelastic X-ray scattering at high pressure and temperature; (e) New CO_2 laser-heated diamond-anvil cell; (f) Absolute temperature measurements; (g) COMPRES environment for automated data analysis; (h) Technical support for dual beam focused ion milling facility for TEM foil preparation; (i) Gas-loading system for diamond-anvil cells at the APS; (j) Development of next generation multi-anvil module for megabar research; (k) Calorimetry-on-a-chip; and (l) Monochromatic X-ray side station at the multi-anvil beamline of the NSLS.

In Part B.2 below, we present detailed progress reports on these Infrastructure Development projects supported by COMPRES in the period 2002-2007. For two of these projects (nos. 2 and 4), program plans and budget requests are presented for the first three years [2002-2010] of COMPRES II.

There are two new initiatives proposed as Infrastructure Development projects for the first three years of COMPRES II: Commission on High Pressure (B.2.m); and a High Pressure Synergetic Center at the Advanced Photon Source (B.2.n). Program plans and budget requests are presented below for these two new projects.

HPSynC would operates on a novel infrastructure concept that connects the large national facility and the high-pressure community. Its main functions would be:

1. To integrate high-pressure techniques with specialized synchrotron beamlines to innovate new capabilities.
2. To integrate high-pressure techniques with general synchrotron beamlines at the Advanced Photon Sources (APS) for high-efficiency high-pressure experimentation.
3. To help the high-pressure Earth materials community to increase the total available beam time by >90 days/yr (in addition to the GSE-CARS beam time).
4. To establish and operate a world-leading high-pressure user laboratory and provide on-site technical support.
5. To introduce new researchers and students to the high-pressure field by organizing training programs and workshops.

(3) Community Activities

Annual Meetings of COMPRES

Each year, COMPRES convenes an annual meeting of the entire community. These meetings will include focus sessions on broad topics of current interest in geophysics and geochemistry, all of which will include keynote talks from prominent members of COMPRES user groups (seismology, geodynamics, mantle petrology, etc.) followed by group discussion, as well as reports from the Community Facilities and Infrastructure Development projects and poster presentations highlighting the most exciting recent scientific achievements.

Workshops and Special Focus Meetings

Over the past four years, 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:

(a) To cultivate and train new users of beamlines at the national laboratories.

(b) To nurture new initiatives which may lead to proposals for leveraged funding.

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 [see examples cited in A.6 above]. We propose to continue this form of partial support in the next five years.

Education and Outreach

In 2007-2012, we plan to continue educational and outreach programs which take advantage of leverage and linkages to other such programs in the Earth Sciences [e.g., EarthScope, IRIS, ESE or its successor].

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 is for a 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 [see details in A. 7 above].

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. These students were chosen from a nationwide competition and pursued independent research projects as well as offering operational support to users at the high-pressure beamlines at the ALS and the NSLS. This pilot experiment proved extremely rewarding for both the interns and their supervisors. We propose to continue this internship program with two positions to be funded each year [1 year maximum duration]. Where possible, this funding will be leveraged by funding from the host national laboratory via their student intern programs.

b. Budget Request for 2007-2012

Previous funding history

COMPRES has been funded in the period May 2002 to April 2007 under a Cooperative Agreement [CAGR] between the NSF Division of Earth Sciences and Stony Brook University [as the host institution for the COMPRES Central Office]. This Cooperative Agreement was based on the original proposal of August 2001 and subsequent review and negotiation.

Current Cooperative Agreement [May 1, 2002 to April 30, 2007]

	Year#1	Year#2	Year#3	Year#4	Year#5	Cum Years #1-5
Orig Request August 2001	$2560K	$2668K	$2728K	$2780K	$2825K	$13561K
Coop Agree	$1800K	$2100K	$2200K	$2300K	$2400K	$10800K
Funded Level	$1967K*	$2200K	$2227K	$2100K	$2100K	$10594K

* includes supplemental funding in Years #1 and 2.

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 be addressed using the facilities operated and the technological developments funded by COMPRES.

Funding request for 2007-2012

In the table below, we present a summary of the budget request for the five-year period from May 2007 to April 2012 organized by the structure and programs of COMPRES:[% of five year total]:

Community Facilities Operations [49%]

Equipment Upgrades [15%]

Infrastructure Development Projects [19%]

Community Activities [4%]

Central Office [13%]

The request of $3245K for Year #1 of COMPRES II [May 2007 to April 2008] is 35% higher than the level authorized by the current Cooperative Agreement for 2006-07. This increase is necessary to ensure that

(1) the operations of the community facilities can serve the rapidly expanding user community.

(2) equipment at the community facilities can receive much-needed upgrades so that the US beamlines will remain competitive (or achieve competitive status) with other national and international laboratories.

(3) new technologies for high-pressure research will be developed for use both at the national laboratories and in laboratories of our home institutions.

(4) workshops which train new users and/or nucleate new funding initiatives can be pursued.

These budget requests are justified in detail in the individual sections of Part B, for both the Community Facilities operations and the Infrastructure Development projects.

The budget requests for Community Activities and Central Office are based on current experience and the program plan outlined above.

BUDGET REQUESTS FOR COMPRES II PROPOSAL
FIVE YEAR TOTALS for Period May 2007 to April 2012
All are in $K and include indirect costs [except where noted]

		Yr #1	Yr#2	Yr#3	Yr#4	Yr#5	5Yr Total
COMMUNITY FACILITIES OPERATIONS/EQUIPMENT UPGRADES							
ALS-West Coast [B.1.a]		Ops	$295	$352	$362	$385	$397
	Equip	$60	-----	-----	-----	-----	$60
X-ray DAC at NSLS [B.1.b]	Ops	$400	$422	$441	$439	$459	$2161
	Equip	$233	$222	$215	$207	$18	$895
IR-DAC at NSLS [B.1.c]	Ops	$282	$297	$312	$327	$342	$1560
	Equip	$80	$230	$100	$80	-----	$490
Multi-Anvil-NSLS [B.1.d]	Ops	$400	$417	$434	$453	$472	$2176
	Equip	$90	$100	$280	$302	$90	$862
Neutron Studies [B.1.e]		$30	$30	$30	$30	$30	$150
Beamline User Housing[a]		$40	$40	$40	$40	$40	$200
Beamline Interns (2)[b]		$127	$132	$137	$142	$147	$685
Subawards (1) IDC		$8	-----	-----	-----	-----	$8
Totals Community Facilities	Ops	$1582	$1690	$1756	$1816	$1887	$8731
	Equip	$463	$552	$595	$589	$508*	$2707

[a]Funds are requested to provide dormitory accommodation for users performing experiments at the COMPRES-supported beamlines at the NSLS and the ALS.

[b]Funds are requested to continue the beamline internship program with two positions to be funded each year [1 year maximum duration]. Where possible, this funding will be leveraged by funding from the host national laboratory via their student intern programs.

	Yr #1	Yr#2	Yr#3	Yr#4	Yr#5	5Yr Total
INFRASTRUCTURE DEVELOPMENT PROJECTS						
Multi-Anvil Cells [B.2.b]	$107	$101	$104	-----	-----	$312
Inelastic X-ray Scatt B.2.d]	$73	$75	$77	------	------	$225
HPSynC B.2.n]	$300	$300	$300	------	------	$900
Comm High P B.2.m]	$30	$30	$30	$30	$30	$150
Workshops (5-7/year)	$100	$100	$100	$100	$100	$500
Subawards (3) IDC	$25	-----	-----	-----	------	$25
Totals Infrastructure Development	$635	$606	$611	$700**	$860**	$3412**
COMMUNITY ACTIVITES						
Annual Meeting	$80	$80	$80	$80	$80	$400
Travel for committees	$30	$30	$30	$30	$30	$150
Education-Outreach	$25	$25	$25	$25	$25	$125
Totals Comm Activ	$135	$135	$135	$135	$135	$675

CENTRAL OFFICE

	Year#1	Yr#2	Yr#3	Yr#4	Yr#5	5Yr Tota
Salary & Fringe Benefits	$382	$416	$411	$447	$484	$2140
Materials	$35	$35	$35	$35	$35	$175
Travel	$13	$16	$19	$22	$25	$95
Totals Central Office	$430	$467	$465	$504	$544	$2410

TOTAL COMPRES II PROPOSED BUDGET

		Yr #1	Yr#2	Yr#3	Yr#4	Yr#5	5Yr Total
Community Facilities	Operations	$1582	$1690	$1756	$1816	$1887	$8731
	Equipment	$463	$552	$595	$589	$508*	$2707
Infrastructure Development Projects		$635	$606	$610	$700**	$860**	$3411**
Community Activities		$135	$135	$135	$135	$135	$675
Central Office		$430	$445	$460	$475	$490	$2300
Totals COMPRES II		$3245	$3420	$3562	$3754	$3934	$17915

* Includes funds for equipment upgrades to be assigned later by Executive Committee on the recommendation of the Facilities Committee. Examples include funds necessary for new facilities at NSLS-II.

** Includes funds for new infrastructure development projects to be assigned later by Executive Committee on the recommendation of the Infrastructure Development Committee.

As per agreement with the Office of Sponsored Programs of the Research Foundation of the State University of New York, these are calculated at the hybrid rate of 33.25% based on a distribution of work on-campus [25% at 55.0%] and off-campus [75% at 26.0%].

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 are being prepared for submission 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

Computational mineral physics—future prospectus

As COMPRES has indicated from the outset of the planning of the organization in early 2001, research in theoretical and computational mineral physics is of fundamental importance to the field of mineral physics and chemistry and to the mission of COMPRES on behalf of the community. This imperative was also clear at the 2003 Workshop in Miami and in the Bass report in 2004:

Computational mineral physics is currently underrepresented in the COMPRES portfolio. Given the rapidly developing capabilities of computational techniques, this is unfortunate, and COMPRES is seeking ways to nurture and develop theoretical initiatives. This is an approach to addressing mineral physics problems that is receiving ever-increasing attention and visibility, both in the U. S. and overseas. This is also an arena in which there are many opportunities for interplay between theory and experiment.

We would like to add computational mineral physics to the overall COMPRES portfolio. Toward this end, the Executive Committee has invited colleagues in this field to submit a brief proposal for a workshop to bring together people from throughout the mineral physics community in the U. S. who have an interest in such a facility and the need for the resources that it would provide. Such a workshop could even be convened in 2006-07 from funds which the ExComm has set aside for workshops leading to new funding initiatives. As with other COMPRES initiatives, we would like to ensure that the community buy-in is as broad as possible, and that there is an opportunity for the community as a whole to contribute input, ideas and feedback. Based on the outcome of the workshop, we hope that together we can evolve a strategy to seek funding for this initiative via a variety of channels

PROCEDURES AND PLANNING FOR COMPRES RENEWAL PROPOSAL

2005

June 19	Open Forum at Annual Meeting in Mohonk, NY
Dec 6-8	Breakfast Meetings: Standing Committees/Executive Committee
Dec 8	Town Hall Meeting at AGU

2006

Jan 26	Issue call for submission of "One-Pagers" highlighting the science and/or technology projects pursued with the support of COMPRES facilities or utilizing data produced at COMPRES facilities.
Jan 30	Issue call for new or renewed initiatives for Infrastructure Development projects, Community Facilities Operations, and Workshops. Deadline: 15 March 2006
March 15	Deadlines for proposals for new or renewed initiatives for (1) Infrastructure Development projects (2) Community Facilities operations. (3) Workshops
March 22	Deadline for One-Pagers highlighting scientific progress
April 15	Executive Committee to receive reports and recommendations from the Infrastructure Development and Facilities Committees.
May 15	Executive Committee to approve program plan and budget for 5-year renewal proposal and submit to President for formulating a draft proposal for the NSF.
June 9	DRAFT of proposal and budget plan to be presented to the Executive Committee
June 16	Revised proposal and budget plan to be sent to the Electorate and the COMPRES community.
June 20-23	5th Annual Meeting at Snowbird, Utah: Presentation of plan and strategy for proposal and discussion with Electorate.

August Proposal for COMPRES renewal to be submitted to NSF-EAR via FastLane.

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PART B: Program Reports

1. Community Facilities Operations
[Report on progress in 2002-2006 and plans for 2007-2012]

2. Infrastructure Development Projects
[Report on progress in 2002-2006 and plans for 2007-2012 for those projects which are slated for continuation]

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
Funding requested for 2007 - 2012: $1791K for operations
$60K for equipment

Summary

We propose to continue to enable COMPRES users to produce the best possible science at the Advanced Light Source (ALS) through: provision of user facilities optimized for their needs, a comprehensive research infrastructure, high-quality personalized user support and dedicated beam time. We also propose to expand the experimental facilities available to COMPRES users to include single crystal and spectroscopic measurements. To achieve these goals we require continued support for the two beam line scientists already in place, support for a post-doctoral level scientist, funds for expendable materials and two new heating lasers. Longer term goals include the development of facilities at the ALS and the Linac Coherent Light Source for x-ray measurements from materials under shock loading.

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. Details of beamtime requests, usage and a list of our COMPRES users given in the Appendices.

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 $Cd_{80}Hg_{20}$ 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.

2. Operation and development plan for the next five years

We are privileged to possess a brand new state of the art laser heating diamond anvil cell facility on beamline 12.2.2. We propose to spend the next five years primarily exploiting this facility in order to achieve the general scientific goals of the COMPRES community. In order to fully focus on beamline 12.2.2, we propose to reduce our support of the systems on beamlines 11.3 and 1.4 to a minimal level, while still maintaining the capability in case of shifts in future user needs and also to accommodate some of the overflow from beamline 12.2.2 when necessary. Key issues include reliability of temperature and pressure measurement under the conditions of the lower mantle. We have a program of validation of our laser-heating system in place and we propose to expand that process in collaboration with the other laser-heating units in the US and elsewhere in order to ensure a high level of accuracy, precision and consistency of measurement between these sites.

We propose to assemble and commission the single-crystal and spectroscopy systems and offer them as facilities for COMPRES users. Initially, the Brillouin system will be operated off-line in the high-pressure laboratory with resistively heated diamond anvil cells. We would aim in the future to combine this system with off-line laser heating and then, depending upon resources and user demand, move the whole system on to beamline 12.2.2. The Raman system will also be assembled in the high-pressure laboratory and made available for COMPRES users there. This is an important addition to our facilities because it allows characterization of poorly diffracting (e.g. amorphous) phases that would otherwise not be well characterized by x-ray diffraction and it offers an important means of independently determining temperatures which is cruscial for the cross calibration described above. Raman is, for many users, the primary method that they use in their home laboratories for characterizing materials in the diamond anvil cell. Having a system at the ALS will provide these users with an important cross-check on the condition of their sample. The system will also be of use to users who do not have their own home systems.

The proposed single-crystal development is in two parts. Firstly, we are developing use of polychromatic diffraction to study crystal structures of materials while they are being laser heated. We have all of the necessary hardware and software in place for this development, and hope to make it available to users by January 2007. Secondly we propose to install a suitable goniometer on beamline 12.2.2 to allow the determination of the best possible high-pressure crystal structures using monochromatic single crystal diffraction. This will extend the maximum pressure that we can determine crystal structures from about 20GPa to between 50 and 100GPa.

The most important national accelerator development at the moment is the LCLS currently under construction at the Stanford Linear Accelerator Center. This source, which will come on line in 2010, will deliver 120fs pulses of x-rays on demand and of sufficient brightness to allow an entire diffraction pattern to be collected from one pulse. This will allow us to collect x-ray data of similar quality of those currently collected from samples held under static pressures in a diamond anvil cell, but at the extreme pressures and temperatures achieved under shock loading. We are currently planning a facility for these measurements as part of the Warm Condensed Matter Group at LCLS and developing a demonstration facility at the ALS for test of principle and initial systems development in collaboration with groups from LLNL and LANL. Further details are given below.

3. Necessary resources

The major risk to delivery of the COMPRES program at the ALS is a potential breakdown of our heating laser. We propose to replace our current heating system with two solid-state lasers. The current laser will be installed on our off-line Brillouin system and moved back into the 12.2.2 hutch at short notice if a breakdown occurs. This is our only request for capital equipment funds and would be most effective if made available in the first year of the renewal.

We also require continued support for our beamline scientists. They are absolutely essential for delivery of the COMPRES program. At the moment they are very overworked and hardly able to cope with the continuous stream of COMPRES users let alone develop their own research programs or new experimental systems. This situation is not sustainable in the long term. We propose the provision of an additional staff resource in the form of a post-doctoral level scientist in order to address this problem. We envisage that this person would spend about half of their time working on the beamline or in the high pressure laboratory supporting COMPRES users. The other half of their time will be devoted to developing their own research projects which will include a large element of collaborative research. We understand the concern that post-doctoral scientists be given full freedom to expand their intellectual horizons and not be burdened with serving a user community. Indeed we have a strong record of identifying and mentoring young scientists. Because of the proximity to the UC Berkeley Campus, however, we believe that it is possible to create a position that has access to a wide diversity of top-quality research groups and facilities, so as to make up for a part-time commitment to serving the user community. We seek a post-doctoral position with limited tenure rather than a permanent staff position, in order to ensure new ideas coming into our program. With adequate checks and balances, we believe that this can be a win-win situation involving a new kind of position for young scientists. As the post-doc becomes familiar with operating our facilities our beamline scientists will gain the quality time required to develop all of our current hardware into fully functioning user facilities and to develop their own research interests. Having research-active scientists running our beamlines is absolutely necessary for us to maintain our capability at the very cutting edge of science. Without the additional staff position it is very unlikely that we will be able to develop either the single-crystal or spectroscopy systems to the point where they are available to general COMPRES users. Also, reducing the workload on our two beamline scientists will reduce the potential for staff turnover, which would greatly disrupt the delivery of the COMPRES program at the ALS.

Now that we have entered into the operations phase of our ALS facilities we will require a slightly higher level of support for expendable materials due to the higher level of COMPRES users conducting experiments at the ALS. We provide all of the equipment necessary for high-pressure, high-temperature research using diamond cells including the diamond-anvil cells themselves. Based on our experience over the past two years we estimate that we consume about six pairs of diamonds and 5 bellows per annum plus gasketing foils. The suite of diamond cells that we have at the moment are in continual use by COMPRES users. This is leading to substantial wear and general degradation. We propose to replace these cells at the rate of two a year for the second through fifth. years of the renewal. This will ensure that we have state-of-the-art diamond cells ready for COMPRES users throughout the renewal period.

No resources are being requested for the shockwave developments. We plan to support this initiative through other DOE and NSF funding lines. COMPRES scientists are encouraged to join us in this work but we feel that a request of funds to COMPRES is premature given the timescales for development of a user facility. We would hope that this will be a major theme of future COMPRES renewals.

4. Cost estimates

Our major proposed expenditure is in salaries. The requested budget contains projected salaries over a five year period for both of our two beamline scientists and an additional mid-band post-doctoral scientist. Estimated costs for diamond cells, diamonds and other expendable materials are contained in Appendix 12. The only capital investment, new heating lasers, is scheduled in the first year in order that the community gets maximum return on this investment.

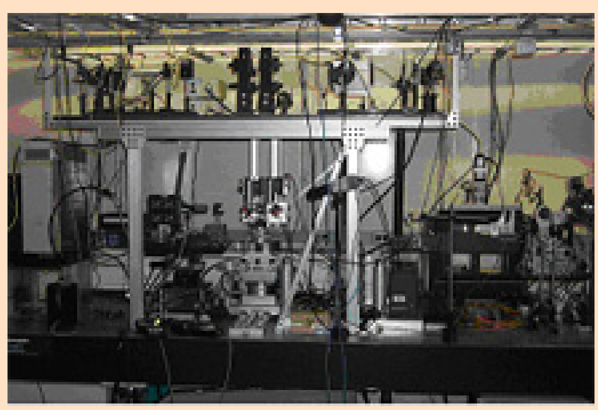
5. Deliverables

In return for this investment general COMPRES users will receive, subject to periodic review by the ALS science advisory committee, guaranteed access to beamline 12.2.2 at about the 25% level and guaranteed access to beamlines 1.4 and 11.3 at the 20% level in addition to the guaranteed access already available to COMPRES users through other approved programs and the general user program. COMPRES users will receive personalized support from the COMPRES beamline scientists and access to the off-line cell loading, sample preparation and spectroscopy facilities. COMPRES will also receive a written yearly activity report and budget request, a presentation at the annual COMPRES meeting and an annual hosted facility visit for the COMPRES Facilities Committee. Also, any other requests for information or general input will be met as quickly as possible given any pre-existing commitments and work schedules.

6. Concluding remarks

COMPRES funding has enabled, for the first time, dedicated user facilities for high-pressure, high-temperature research on the west coast of the US. These facilities are now operating and being exploited by COMPRES users. Demand for these facilities is high but COMPRES users have been successful in securing the majority of the available beamtime for their research. Exploiting these facilities is now our main focus. Continued financial support is requested in order to provide personnel and expendable materials in support of the COMPRES program and to provide additional hardware that is needed as insurance against critical (single-point) systems failure. The facilities at the ALS are truly state of the art being newly constructed and commissioned. We have a great user support team in place. In partnership with COMPRES we expect to be able to complete the link between seismology and mineral physics resolve major outstanding issues of the upper and lower mantle next five years. We will also lay the ground work necessary for our community to then move on to achieving a detailed understanding of the core and other planetary interiors.

Summary of current West Coast COMPRES facilities



Beamline 12.2.2 at the ALS is a dedicated high-pressure beamline made possible by COMPRES facilities support

Total Budget Request

	2007	2008	2009	2010	2011
Total Salaries	180,732	192,231	197,998	212,045	218,407
Benefits (17%)	30,724	32,679	33,659	36,045	37,129
Travel	6,000	8,500	9,000	9,500	10,000
Expendable materials	12,246	51,007	52,537	54,113	55,736
Equipment	60,000	0	0	0	0
Total direct costs	294,127	238,810	246,057	262,992	270,936
Indirect costs (26%)	60,873	62,091	63,975	68,378	70,443
Total costs	**355,001**	**352,001**	**362,000**	**385,000**	**397,000**

Detailed cost of expendable materials

		2007	2008	2009	2010	2011
Metals		2,046	2,107	2,171	2,236	2,303
Diamonds		10,200	10,506	10,821	11,146	11,480
Bellows			8,455	8,709	8,970	9,239
Diamond cells	Number	0	2	2	2	2
	Unit cost		14,969	15,418	15,881	16,357

COMPRES supported facilities on the West Coast are currently focused at the ALS, and consist of:

Beamline 12.2.2 (Diffraction and imaging with laser heating). This is a hard x-ray (8-35keV) focused synchrotron beamline situated on one of the superbend sources at the Advanced Light Source. It contains two end stations. End station 1 has a large x-ray focal spot (100 x 80 µm) and is equipped for x-ray diffraction measurements from powders held in resistively heated diamond anvil cells using a Bruker CCD detector. End station 2 has a smaller focal spot (10 x 10 µm) and is equipped with a double-sided laser-heating system, ruby fluorescence system and Mar345 image-plate system. Both endstations are equipped with sufficient goniometry to allow automatic alignment and setting of the sample to detector distance. End station 1 is currently being upgraded with additional goniometry to allow high-pressure single-crystal diffraction measurements. End station 2 is equipped to allow radial diffraction studies and is being modified to allow laser heating of samples during these measurements. It is also equipped with an x-ray imaging system. This allows x-ray shadow graphs to be taken of the contents of diamond anvil cells while heating resistively or with the laser. This is used for falling sphere viscosity measurements and studies of assemblage reactivity. Both end stations are equipped with automated gas delivery systems to allow remote pressure control. In terms of diffraction and imaging this beamline is equivalent to any in the world for a mineral sample of average x-ray scattering power. Laser heating in radial geometry and an integrated imaging system for viscosity measurements are only available at the ALS.

This is our primary high-pressure resource at the ALS and we aim to focus our resources on exploiting this beamline over the next five years.

Beamline 11.3 (Diffraction). This is a medium energy synchrotron x-ray beamline which benefits from x-rays from one of the ALS bending magnets. It was original developed as a temporary platform to allow us to make high-pressure x-ray diffraction measurements while beamline 12.2.2 was under construction. It is equipped to allow high-pressure diffraction measurements from samples contained in diamond-anvil cells using a Bruker CCD. Because this beamline is located on a bending magnet the maximum x-ray energy is limited to 17keV. This makes the beamline very useful for looking at samples with long lattice repeats for example hydrous minerals. This beamline is shared with Chemical Crystallography. Usually high-pressure gets about 25% of the available beamtime.

The usefulness of this beamline is limited but it is working routinely and needs little maintenance. We plan to reduce our support of this beamline for high-pressure measurements over the next five years but maintain the infrastructure should we need it in the future and to accommodate any experiments that do not absolutely require the additional benefits of beamline 12.2.2.

Beamline 1.4 (Infrared). This is a state of the art synchrotron infrared beamline situated on one of the bending magnets at the Advanced Light Source. It is equipped with a fully automated Thermo Nicolet Nexus 870 FTIR and has a diffraction limited focal spot (3-10μm). Measurements are currently made using the standard end station microscope as a mount for the diamond cells. This does not allow every design of diamond cell to be used on the beamline. Two low-profile diamond cells suitable for working with this end station are available for COMPRES users. One is equipped with an external resistive heater. This is a shared beamline with about 20% of the time available for high-pressure studies.

High-pressure infrared data from this beamline are of as high a quality as that from any other beamline in the world. The main drawback is that only special low profile diamond cells can be used with the current experimental arrangement. This may have led to the low demand for this facility. We plan to reduce support of high-pressure work on this beamline over the next five years but maintain the infrastructure should we require this resource in the future. If demand for this beamline should grow then we could rearrange the experimental layout to allow any design of diamond cell to be used.

High-Pressure Laboratory. The high-pressure laboratory contains all of the equipment necessary to align and load diamond-anvil cells. This includes microscopes, spark eroder, micro-drill, cryogen loading, gas delivery systems to drive bellows for indentation, etc. The laboratory also contains two optical tables upon which we are currently assembling Brillouin and Raman spectroscopy systems. This laboratory is available for COMPRES users for cell and sample preparation and is equipped with an automated falling sphere viscosity measurement system suitable for transparent samples.

Off-line ruby system. This system is situated close to beamline 12.2.2. It has a gas delivery system to allow pressurization of bellow driven cells. Diamond cells are mounted on a microscope stage that allows samples to be viewed using reflected and transmitted light. Use of the microscope limits the range of diamond cell design that can be used. We are currently upgrading this system to a more general design that can accommodate any type of diamond cell.

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S. Speziale and C. E. Tommaseo (2006). Diamond anvil deformation experiments in radial diffraction geometry. J. Phys. Cond. Matter.ALS

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

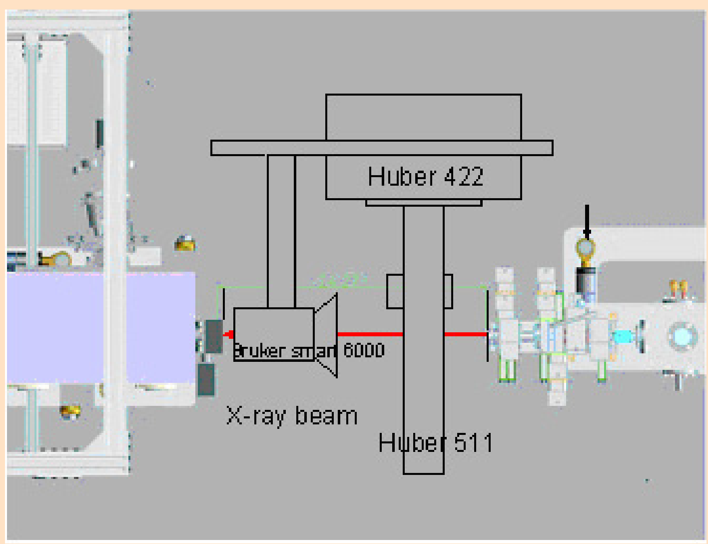
1. **COMPRES laser heated diamond anvil cell workshop.** Organisers: Simon Clark and Michael Walter. 22 February 2003.
2. **IUCr/COMPRES sponsored high-pressure practicum**. Organiser: Simon Clark. 4 December 2003.
3. **I*UCr/COMPRES sponsored workshop:* High Pressure Structure and Reactivity: The Science of Change.** Oganisers 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.

Potential Single crystal diffraction facility at the ALS

The very first x-ray diffraction experiments at high-pressure were performed in diamond anvil cells on sealed-tube sources using a single-crystal as sample. This intrinsically limited the maximal pressures obtainable to pressures below 20 GPa, mainly due to the minimal sample size required to extract useful intensities from the sample. Thanks to the high brilliance of third-generation synchrotron radiation, this limitation could be overcome with the help of dedicated high-pressure beamlines at the ESRF and APS. Single-crystal diffraction was largely abandoned at high-pressure beamlines and replaced by powder diffraction, at least in part because of an understandable focus on phase transformations in complex (multi-component) systems simulating the Earth's interior.

Powder diffraction, however, has intrinsic limitations when trying to push the limits to higher pressures and temperatures. The most dramatic limitation arises from the smaller and smaller sample volumes necessary to achieve extreme conditions of pressures. This together with sample recrystallization at high-temperatures unavoidably leads to a significant reduction in powder grains in the x-ray beam thus degrading the powder statistics. Thus, powder diffraction is not the uniquely ideal tool for high-pressure crystallography.

This problem can be circumvented by re-establishing single-crystal diffraction at synchrotron high-pressure beamlines, now possible because modern 3rd generation synchrotron sources have beam position stabilities that are more than sufficient for single-crystal diffraction. Moreover, in cases where sample movement is restricted or



Schematic diagram of high-pressure single crystal system being installed in beamline 12.2.2

impossible, the high fidelity of modern synchrotron monochromators allows one to scan in energy rather than in angle, and thus use a polychromatic approach to scan the reciprocal lattice through the Ewald sphere. This polychromatic technique is to a large

extent complementary to monochromatic techniques: Monochromatic single-crystal diffraction at a synchrotron beamline will allow us to extend the pressure range at which high-quality structural data can be obtained from 20 GPa in homelabs to 50 – 100 GPa. This opens up a large range of crystal chemical problems that can be tackled with diffraction methods (e.g. analysis of displacement parameters, subtle structural distortions in framework structures etc.). Polychromatic techniques, on the other hand, can be combined with laser heating and thus for the first time will enable extracting crystal-structural details of materials at conditions of the center of the Earth.

The need for synchrotron-based high-pressure single-crystal diffraction in the US has been recognized by the community. In November 2004, a workshop on this topic was organized in Chicago, and its results published as a special issue of the *Journal of Synchrotron Radiation* (September 2005). Another outcome of this workshop was the formation of a project group consisting of Malcolm Nicol (University of Nevada), M. Bonner Denton (University of Arizona), Przemyslav Dera (Geophysical Laboratory, Washington), Robert T. Downs (University of Arizona), Mark Rivers (University of Chicago) and Martin Kunz (ALS). The group successfully submitted a proposal to the NSF-MRI (major research instrumentation) program. The aim of the project is to coordinate a concerted effort to establish a high-pressure single-crystal diffraction program at the NSLS, APS and ALS. New techniques (polychromatic single-cystal diffraction) shall be developed, and mature techniques (monochromatic high-pressure single-crystal diffraction) implemented. The project was awarded $712,925 for personnel and hardware, with $50,000 allocated to ALS to help the high-pressure group acquire a suitable single-crystal goniometer. The new single-crystal goniometer is planned to replace the current 1-axis goniometer on end station 1 of beamline 12.2.2. Beamline 12.2.2 offers the advantage that both white-beam and monochromatic measurements can be conducted in this hutch. The big disadvantage is that the beamline is already commissioned for laser-heated diamond-anvil cell experiments, and is heavily oversubscribed and may not be able to fulfill the needs of the COMPRES community for this technique.

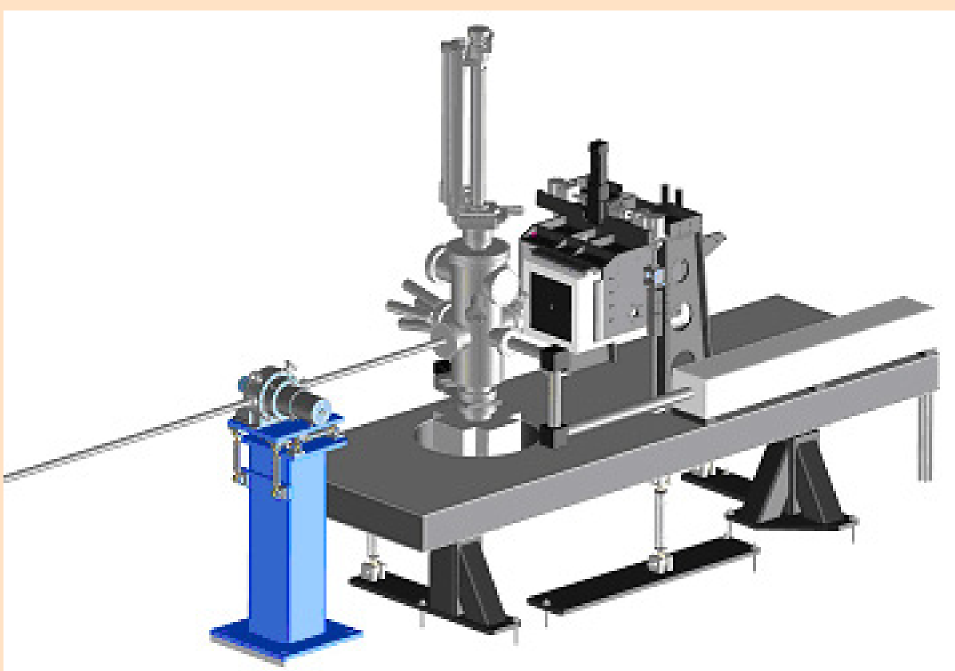
Plans for a Shock-wave facility

The traditional method used to generate high-pressure and temperatures for diffraction measurements involves using a diamond-anvil cell and resistive or laser heating. Single-crystal x-ray diffraction data have been collected up to combined temperatures and pressures of about 10-20 GPa and 1000^{o} C. The maximum temperatures achievable using a diamond cell can be extended to a few thousand Kelvin by using laser heating. This is routinely done using polycrystalline samples, but has not yet been successfully applied to single crystal samples. One reason for this might be the very large temperature gradients that are present using laser heating, causing strain in the single-crystal sample and low-quality diffraction patterns.

An alternative method of pressure and temperature generation involves using shockwaves. Two methods of shockwave generation are commonly used. In the first a metal plate or flyer is propelled into the sample using either an electromagnetic field or rapid expansion of a gas. This commonly involves a large sample (~cm diameter and ~mm thick) but the impellor mechanism is large, may take many hours to rearm ready for the next measurement, and is not suitable for combining with current synchrotron beamlines. In the second, a laser is used to ablate a small portion of the sample or a material coated on the surface of the sample. The force generated in opposition to the expansion of the ablated material passes through the sample and generates a shockwave. This method typically uses a sample size of about 1 mm diameter and 10 ìm thick and has a repetition rate, that varies greatly with laser system, of about 10^{-3} to 10 Hz.

These methods have been used to generate combined pressures and temperatures in the hundreds of GPa and thousands of Kelvin, and overcome the limitations of the diamond cell. They do have their own limitations, however, including: samples suffer from possible preheating effects and difficulty in ensuring constant pressure, and the experiments are difficult to synchronize with diagnostics. Still, an advantage of using shockwaves to generate high pressures and temperatures is that the velocity of the shock can be measured using Doppler velocimetry, such as Visible Interferometry System for Any Reflector (VISAR). This allows the pressure to be accurately determined, and the temperature to be measured by spectroraidometry, given adequate equations of state for internal standards. As such this method would seem to overcome the inherent limitations of the diamond-cell technique, but the shocked state is only transient with a stable temperature and pressure lasting for about 1µs for a gas gun and about 10 to 100 ns for a large laser. Also, the sample is generally destroyed by the shock.

X-ray diffraction measurements from samples in the shocked state actually date back to the 1970s, with a series of pioneering experiments performed at the Lawrence Livermore National Laboratory. This capability has been developed over the years, and state of the art systems now allow x-ray diffraction measurements with a temporal resolution of a few nanoseconds or less. Temporal resolution can be achieved by using a short x-ray pulse, and/or time resolved diagnostics such as an x-ray streak camera. These devices typically have an aperture of only a few degrees, which allows only a limited volume of reciprocal space to be sampled during each shock event. Given the variations in pressure and temperature inherent when using shockwaves to generate high pressures and temperatures, a much more satisfactory arrangement would be to collect all of the necessary reflections simultaneously or with the least number of measurements possible. Ideally one would want to achieve the necessary



Schematic diagram of planned shockwave setup on beamline 5.3.1

temporal resolution by using a single pulse of x-rays having a short time duration. That way, one could use a conventional area detector such as film, imaging plate or CCD detector, and sample a large volume of reciprocal space simultaneously.

New free electron-laser sources of x-rays being developed in Europe, Japan and the USA will allow us for the first time to be able to collect entire diffraction patterns using 100 fs pulses of x-rays. The source being constructed in the US, the Linac Coherent Light Source, is situated at the Stanford Linear Accelerator Center in the South Bay and will come online in 2011. COMPRES scientists are already involved in the planning of the experimental facilities that will be available on this source, and one of the five first facilities to be developed will include apparatus for our shockwave measurements. We envision that COMPRES will play a key role in developing and supporting these facilities as they come on line, but due to the time line that will be the subject of future COMPRES renewals.

One of the key scientific questions to be addressed with such capability regards the structure and bonding in fluids at deep-planetary pressures. Silicate melts, for example, are expected to undergo structural transformations under pressure, analogous to the polymorphic transformations of crystalline phases, the result being that melts tend to be comparable in density to their co-existing solids at high pressures. This is important because the effect of volcanism at shallow-planet conditions, to separate (differentiate) compositionally distinct regions of a planet and to rapidly move heat by fluid motion, is completely altered at deep-planetary conditions. That is, “anti-volcanism” with melts sinking rather than rising toward the surface may characterize deep interiors, implying a long-term retention of heat at depth that is not seen at shallow conditions. In addition, there are major questions regarding the properties of fluids at conditions achieved inside giant and super-giant (exo-) planets, and also during the giant-impact phase thought to characterize the late stages of planetary birth, that can be addressed for the first time by means of the proposed development of experimental capabilities.

What we need to do in the short term is to develop the necessary experimental skills and expertise to allow us to exploit the LCLS and related facilities when they come on line. Currently, beamline 5.3.1 at the ALS is being equipped with the necessary laser to generate shockwaves and x-ray shutter to allow us to start this development program, to collect some of the first data and to address some Earth sciences issues.

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]

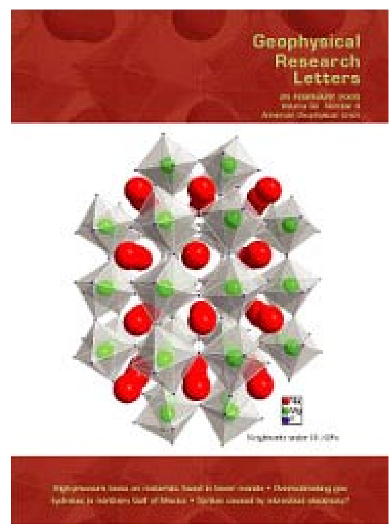
New Operation Team starting May 2007:
Stony Brook University [J. Chen], Princeton University [T. S. Duffy], University of Chicago [M. L. Rivers], and Carnegie Institution of Washington [A. Goncharov]

Curent funding for 2002-2007: $1074K
Funding requested for 2007-2012: $2161K for operations and $895K for equipment

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 $NaMgF_3$: 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 $MgSiO_3$ 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 $NaMgF_3$ 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 $NaMgF_3$ 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 octa-hedral tilting matches that of the bond length compression. This is followed by an increasing contribution from the octa-hedral 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 Fe_2Si. 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.

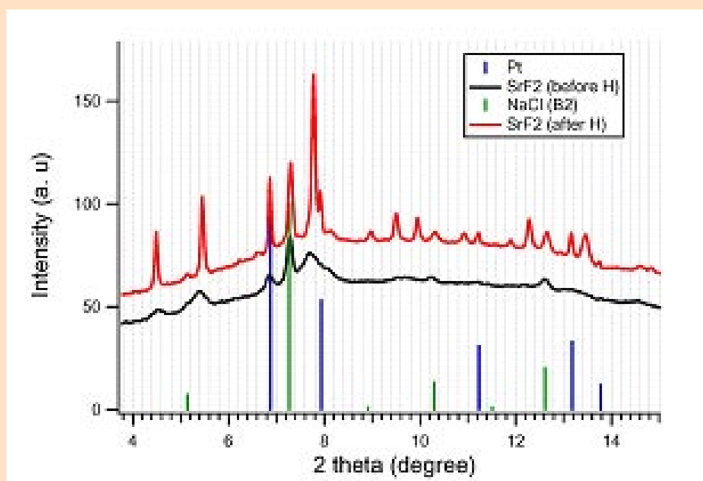
Strength and Elasticity of SiO_2 across the Stishovite - CaCl-type Structural Phase Boundary: Stishovite is known to transform to orthorhombic $CaCl_2$-type structure at 50±3 GPa. The study of elastic instabilities is important for understanding phase transformations, and the stishovite–$CaCl_2$-type 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 SiO_2 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 $CaCl_2$-type phase transition boundary is approached. The differential stress then recovers rapidly to values of 5±2 GPa at 52-55 GPa in the $CaCl_2$-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 Å, b = 5.044 Å, c = 9.500 Å, and its unit cell volume was 416.4 Å^3, which was about 2 % smaller than the unit cell volume of gibbsite at ambient conditions. The second order Birch EoS fitting for the high pressure phase yields bulk modulus of 75

± 2 GPa based on the assumption of $K_0' = 4$. The high-pressure phase also showed partial disordering as diffraction peaks broadened. To understanding the broadening, they also performed *in-situ* high-pressure infrared absorption spectra experiments at beamline U2A. From its broadened IR vibrational modes above 15 GPa, while the Al-O substructure still kept stable from the corresponding XRD data. A gradual disordering of hydrogen substructure above 15 GPa in a quasi-hydrostatic compressing is suggested. The disordering of hydrogen may induce a small amount of local disordering in the Al-O basic structure, but is insufficient to drive the system to complete amorphization under compression. The further broadening of XRD patterns above 30 GPa demonstrated some extent the disorder distribution of the Al-O substructure, but they still remained the "crystalline" instead of complete amorphization within the experimental range up to 53 GPa.

Constraining the equation of state of fluid H_2O 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 H_2O 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 $K_{T0} = 21.1 \pm 1.3$ GPa, its pressure derivative $K'_{T0} = 4.4 \pm 0.1$ and the volume $V_0 = 12.4 \pm 0.1$ cm^3/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 H_2O at elevated pressures and temperatures by taking the pressure derivative of the Gibbs free energy difference between Ice VII and fluid H_2O along the Ice VII melting curve. Comparison of these results suggests that the previously stated equations of state of fluid H_2O overestimate the molar volume of fluid H_2O at pressures greater than 20 GPa.

Phase Transition in SrF_2 by laser heating at X-17B3: There has been considerable interest in understanding phase transition sequences in divalent metal fluorides AF_2 (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 SrF_2 using the laser-heated diamond anvil cell at beamline X17B3 at the NSLS. Pure SrF_2 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 BaF_2 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



Diffraction patterns obtained at X17B3 from SrF_2 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.

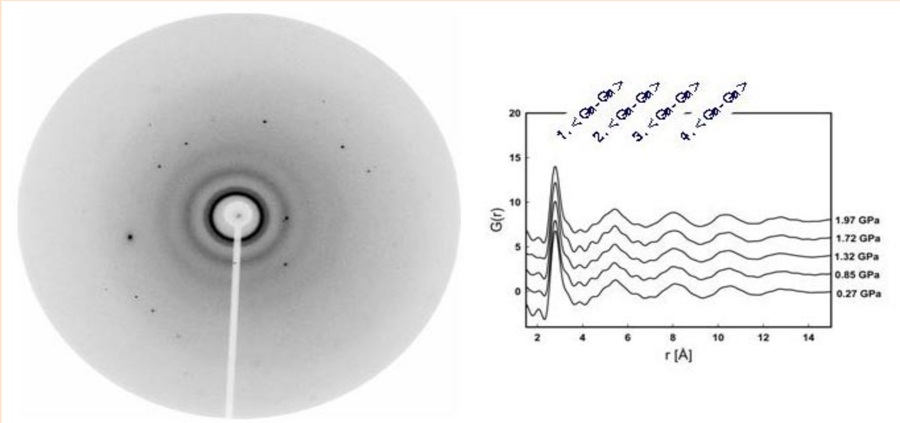
patterns and further experiments to constrain the phase boundary.

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), $Ca_{10}(PO_4)_6(OH)_2$, 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, $Ca_{10}(PO_4)_6(OH)_2$ 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 indicate that the 1st and 2nd nearest neighbor bound lengths are insensitive to pressure whereas the 3rd and 4th nearest neighbor bound lengths are more sensitive to pressure. A clustered local structure in the liquid



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

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

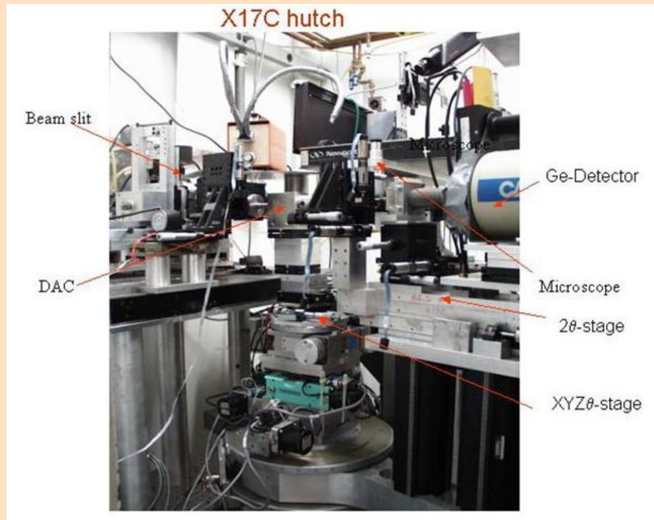
The diamond anvil cell X-ray (DAC-XR) facilities at the National Synchrotron Light Source (NSLS) are located on a superconducting wiggler beamline and consist of two stations (X-17C and X-17B3) together with a sample preparation laboratory. The DAC-XR facility is one of the longest-running high-pressure 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 with 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-2θ 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 sagitally 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.

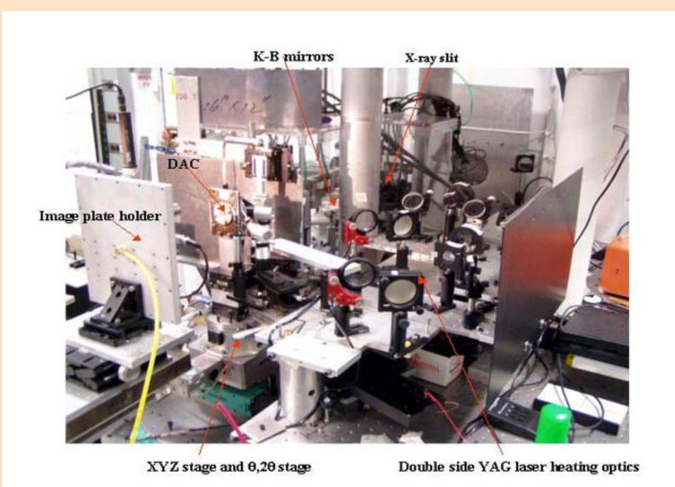


X-17B3:

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 results 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 ~10 x 20 μm in size. A double-sided laser heating system using a Nd:YAG laser has now beeninstalled 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.



and improved clean-up slits. The Kirkpatrick-Baez mirror system will be overhauled with the aim to achieve <10 μm x 10 μm beamsize 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.

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.

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,

II. Development, Operation, and Management Plan (2007-2012)

A. Overview

The objective for the next phase of COMPRES is to build on the long-term success of DAC-XR at NSLS and to further develop it into a world-leading high-pressure research facility to meet the growing needs of the high-pressure geoscience community. The DAC-XR facility has unique characteristics that make it especially well suited for high-pressure Earth science research: 1) NSLS DAC-XR has a brilliant high-energy wiggler x-ray source. 2) X-17C has unique capabilities for energy dispersive diffraction for which there is high scientific demand and is ideally tailored for the geometry of the diamond anvil cell. 3) The NSLS DAC-XR facility in combination with U2A infrared facilities provides unparalleled capabilities to combine x-ray and spectroscopic (Raman and IR) studies under in situ high-pressure conditions over a wide pressure-temperature range. Overall, the superconductor wiggler X17 delivers the best x-ray beam among the COMPRES-supported high-pressure facilities and the second best among all facilities in the US after the undulator of the APS. In view of the severe oversubscription for the DAC beamtime at GSECARS at the APS, the NSLS DAC-XR facilities with one full-time and one half-time dedicated beamline will continue to serve as a workhorse to meet the community need for in-situ high pressure research.

User input for this plan was solicited and obtained from the NSLS X-Ray High Pressure Research Workshop held on February 25-26, 2006 and attended by ~50 persons. Many others contributed input by email or telephone conversations. The two-day workshop held 8 breakout sessions focusing on science and technique challenges and beamline planning. Summaries of the breakout sessions are available on-line at http://www.mpi.stonybrook.edu/NSLS/XHP/breakoutsession.html, based on which this beamline operation plan is developed.

B. Scientific Opportunities

A large number of current and potential users were asked what technique(s) they would most want to use at the NSLS DAC-XR facility. The overwhelming response was for further development of laser heating capabilities to allow in situ measurements up to Mbar pressures. The demand for laser heating in the high-pressure community is clearly very strong and growing rapidly. Laser heating facilities at other beamlines (e.g. GSECARS, HPCAT) are heavily oversubscribed.

The demand for laser-heating capabilities reflects underlying scientific opportunities. Only the laser heated diamond cell can reproduce P-T conditions of the Earth's lower mantle and core. Recent years have seen the discovery of many important phase transformations such as the post-perovskite phase of $MgSiO_3$. Laser heating experiments also are necessary to probe melting, thermal equations of state, chemical reactions and other high-temperature phenomena. Laser heating also plays a key role to reduce deviatoric stresses, and thus is increasingly critical to control and define the stress state in a wide variety of high P experiments. In the future, many applications will require more uniform temperature distributions that are measured accurately to higher precision than achievable at present. Therefore, laser-heating developments at X-17B3 will focus on improvements to temperature measurement capability.

There also remains strong interest in energy-dispersive diffraction techniques. Single-crystal experiments using the white beam are a powerful method for non-destructive phase identification, crystal orientation, and structure determination of micro-inclusions and high-pressure samples. Energy dispersive capabilities are also very important for liquid/amorphous scattering experiments and there are new opportunities for high-energy experiments as discussed below. Many other experiments such as radial x-ray diffraction measurements can benefit from the spatial filtering afforded by the energy dispersive method. These benefits are reflected in the X17 publication list (see appendix) which contains a high percentage of papers using energy dispersive techniques.

The development of high-resolution

diffraction techniques has revealed the existence of subtle but important phase changes that were not accessible to examination previously. The detailed crystallographic response to compression can be uncovered using whole powder pattern refinement techniques that require high-resolution data and reliable intensity information. Monochromatic experiments also open opportunities for studying local structure of high pressure melts using high energy x-ray diffusion scattering (enabling research for understanding melts in the mantle and core), texture development and plastic deformation using the radial x-ray diffraction. The combination of monochromatic and energy dispersive diffraction and the laser heating capability at DAC-XR will enable a broad spectrum of users to continue to address forefront scientific problems, including new opportunities for single crystal study at high pressures.

Study of rheological properties of minerals at high pressures has been an important task of geophysicists. While the multi-anvil press team at the X17B2 has been playing a critical role in advancing the pressure range of strain / stress measurements to beyond 10 GPa, there still be a large pressure range to explore for flow-law study to enhance our understanding of mantle dynamics. Early studies of minerals' strength using in-situ side-entrance DAC x-ray diffraction were conducted at X17C. Integrating the high pressure rheology expertise at the NSLS is a unique opportunity for challenging the pressure limitation in rheological studies. As a RDA (Rotational Drickamer Apparatus) has demonstrated its great potential in the deformation experiment, we plan to explore the application of a rotational DAC (RDAC) to rheology study. In addition, as the NSLS II will significantly improve the resolution of x-ray imaging to nanometers, direct strain / stress measurement in an automated compression DAC may be possible to produce meaningful data for applying to Earth's interior.

C. Technical Challenges

1. P-T generation and control

The ability to control pressure and temperature are central to any high-pressure beamline and often are a limiting factor in the science to be pursued. Many users have excellent science proposals but do not have access to the appropriate DAC tools. Currently, DAC-XR has only very limited capability to supply diamond cells to users. We propose to make a wide range of diamond cells available and accessible by the user community. This basic set of diamond cells needs to include: symmetric cells for laser heating, Mao-Bell type cell, open access cells (short piston cylinder) for wide two-theta range, membrane and motor-controlled cells for fine pressure control and remote pressure change, and rotational diamond cells. In addition, external heating capabilities are needed which involves cells constructed from appropriate materials as well as furnace and heating assemblies.

The current ruby fluorescence system plays an essential role not only in the X-17B3 and X-17C operations but is also accessed by experiments at other beam lines at the NSLS (e.g. X7). Capability of uninterrupted service of the ruby system is critical to the DAC research at the NSLS. The current air-cooled Ar laser (class IIIb) is more than 1000 hours beyond its expected lifetime and a replacement laser is requested in the budget. We also propose to build a portable ruby system. This system will add on-line pressure calibration capability for x-ray studies at high (external heating) and low temperature and improve operational efficiency for many users. We can build the system in a cost effective manner by sharing the spectrometer and detector between the two systems and using fiber optics to deliver the beam. Separate control computers are required for each system.

The current laser heating system at X17B3 is more than 10 years old and greatly lags other systems in user-friendliness, automation, and overall equipment capability. A complete renovation of the system is necessary including new laser, optics, spectrometers, and detectors. The near-IR (e.g. Nd:YLF laser) remains the most generally useful wavelength range and a new class of diode pumped lasers are now available. Designs incorporating both refractive and reflective optics will be considered. The key point for development of this system is that we plan to focus on improving the quality of the temperature measurements so that high-precision, accurate, user-verifiable temperature measurements can be quickly and reliable obtained under in situ conditions.

A cryostat is needed to expand the capabilities of DAC-XR to low-temperature experiments which are of fundamental interest in many systems of interest to planetary science and condensed matter physics. We propose to obtain a custom cryostat suitable for high-pressure experiments using a variety of different diamond anvil cells. This will allow coverage of the temperature range from 2.2 – 350 K.

2. X-ray beam

As a coherent effort with X-17B2 multi-anvil program, we suggest COMPRES leverage some seed fund to promote the installation of X-17A. Currently four research programs time-share the beam time of X17. As X-17B2 and B3 can run simultaneously, each station has 50% availability of the total beamtime. Installing X17A (mono beam) will increase the high pressure shared time to a possible 80% of total beam time.

X-17C will retain white beam capability, but a rapid and reproducible capability to switch between white and monochromatic modes is essential. Working with NSLS staff, monochromators will be improved to achieve higher flux. The current four-crystal monochromator in X-17B3 significantly reduces flux at the sample and will be replaced. It is expected that we can ultimately achieve monochromatic x-ray intensity nearly one order of magnitude higher than the bending magnet beamline at the APS. For monochromatic experiments, we also plan to achieve energy coverage from 20-100 keV.

For many experiments including the laser-heated diamond cell, a micro beam size of less than 10 μm is required. The K-B mirror system will be upgraded to achieve this on both beamlines. The quality of the x-ray beam shape is also crucial to reduce unwanted signal from the gaskets and other components. A high-quality motorized clean-up slit system will be constructed.

The superconductor wiggler is characterized as high energy x-ray source. Dr. Zhang of the NSLS has developed a two-dimensional focusing monochromator with a characteristic energy of 67.7keV. Experiments at B3 have been conducted at 80keV photon energy. Such a high-energy mono-beam would be highly advantageous for studying structures of noncrystalline materials for a high Q coverage in total x-ray scattering.

3. Detectors

The DAC-XR facility currently relies on a very old off-line image plate system for monochromatic experiments. This system has a number of disadvantages including lengthy processing times and preclusion of in situ (high temperature) x-ray measurements. The quality of the x-ray detector is an extremely important component of an x-ray diffraction experiment. Both imaging plate (IP) and CCD detectors have relative merits for various experiments as the former provides highest resolution while the latter is optimum for rapid in situ measurements. We propose to install modern on-line IP and CCD detector systems for use at the DAC-XR facility. The IP will be a MAR345 or equivalent while the CCD system will be a MAR-165 CCD or equivalent. The MAR345 will be shared between 17C and 17B3 while the MAR-165 CCD will be shared between the diamond cell and multi-anvil facilities.

We have also included a request to purchase two new germanium detectors to replace the existing detectors that are more than 10 years old and whose performance is significantly degraded.

4. Beamline controls

We will upgrade the optical table that supports the diffraction and laser heating system in X-17B3, and upgrade the stages in X-17C.

High-quality sample imaging capabilities are becoming increasingly important at beamlines. Such systems as cooled CCD cameras have numerous advantages including rapid sample alignment and imaging of low levels of x-ray induced luminescence. We will purchase new imaging cameras for both beamlines.

5. High Pressure Laboratory

The high-pressure lab at DAC-XR needs significant upgrades to support the needs of the high-pressure community. High-quality sample preparation is the key for many experiments including laser heating and those involving highly sensitive samples. Many potential users do not have high-pressure facilities at their home laboratory so high-quality equipment and adequate support by beamline personnel is essential. A new sample preparation microscope is needed. A gas loading system based on the APS design will be purchased and installed. Gas loading systems are essential for achieving the most hydrostatic conditions possible (e.g. He pressure medium) and for providing flexibility for a range of rare gas solids as insulation and pressure media (Ne, Ar, Kr). Another major item required at NSLS is a glove box with microscope attachment for sample loading. There is currently no such system available to users at the NSLS.

6. Brillouin Spectroscopy System

We plan to install an on-line Brillouin spectroscopy system in X-17B3 for combined x-ray diffraction and elasticity measurements after there is a track record from the Brillouin operation at the GSECARS beamline at APS and the demand for such facilities installed online at synchrotrons can be assessed. No funds are requested.

D. NSLS-II

NSLS-II is a proposed medium energy (3 GeV) storage ring that will be able to produce x-rays 10,000 times brighter than those produced at NSLS today. Among the expected features of this facility are continuous top-off mode operation, more than 20 insertion devices, and ability to produce nano-sized beams. Construction of this facility is planned to begin in 2008 and operations will commence in 2012. High-pressure research is one of key scientific programs highlighted in the NSLS-II initiative. We recognize that continued support and enhancement of high-pressure x-ray facilities at NSLS will lead to opportunities for new types of experiments at NSLS-II. During the lifetime of this proposal we will be actively involved in planning for a new generation of high-pressure capability at NSLS-II.

F. Management Plan

We plan to institute a new multi-institution management plan where participants from each institution will bring unique capabilities that together will provide an excellent team for development and maintenance of this facility. The management team will consist of Jiuhua Chen (PI) and three co-PIs: Thomas Duffy, Mark Rivers, and Alex Goncharov.

Jiuhua Chen, SUNY Stony Brook. Chen has experience in synchrotron-based multi-anvil and diamond cell experiments. He also has extensive knowledge of beamline development at NSLS. As PI, Chen will provide overall guidance and supervise beamline personnel for the DAC-XR facility. Chen will also serve as a liaison between the diamond cell and multi-anvil programs at X17C. In this way, we will achieve synergy and coordination between the high-pressure groups that could not be achieved otherwise. Chen provides on-site leadership that is essential for proper management and oversight as well as rapid response to both technical problems and scientific opportunities as they develop. Chen will also direct scientific research direction that takes unique advantages at the facility (e.g. high energy melt/glass x-ray scattering) and seek for external funding and supports (e.g. personnel support from SBU and the NSLS) to the facility.

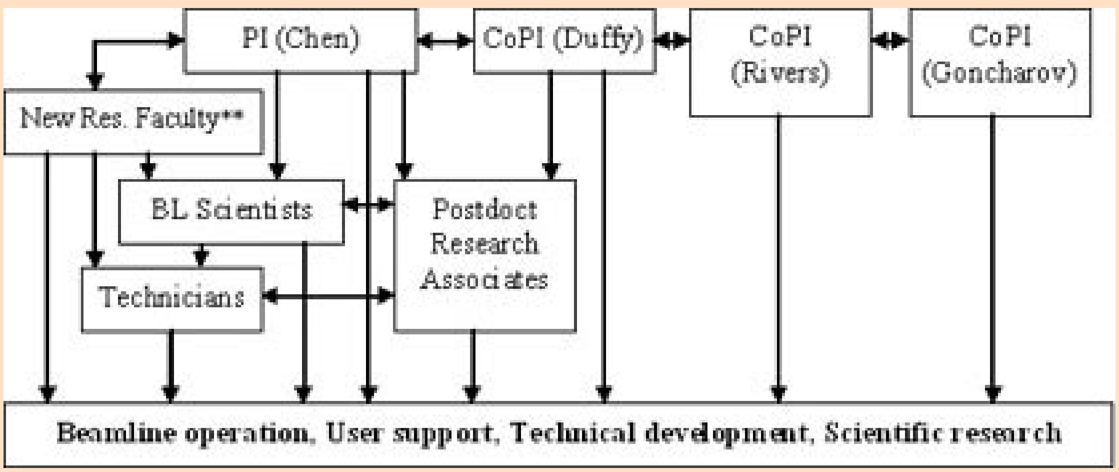
Thomas Duffy, Princeton University. Duffy is a diamond anvil cell specialist and has been a user of the NSLS since 1993. He has experience in a wide variety of synchrotron based high-pressure experiments, and formerly served as beamline scientist at the GSCECARS facility of the Advanced Photon Source. Duffy will coordinate the scientific agenda for the new DAC-XR program, co-supervise beamline postdoc fellows, serve as interface to the user community as well as seek for external funding for facility development and related scientific research,

Mark Rivers, GSECARS, U. of Chicago. Mark Rivers is manager of the GSECARS sector at the Advanced Photon Source which is one of the most productive and successful beamlines at the APS. The University of Chicago has also remotely managed X-26 of the NSLS for many years. Rivers brings the immense technical resources of

GSECARS to help strengthen the NSLS program. This has already been done informally over the years in critical areas such as focusing optics, detectors, and software. GSECARS has unparalleled expertise in these areas and also in mechanical design. The participation of GSECARS also enables development of common tools across beamlines which makes it easier for users to carry out experiments at multiple beamlines. Rivers will thus supply technical expertise to the management team.

Alex Goncharov, Geophysical Laboratory, Carnegie Institution of Washington. Goncharov has extensive experience in optical systems for high-pressure research. He will be intimately involved in the design, testing, construction, and operation of the new laser-heating system at X17B3. He also will serve as a liaison between the x-ray and infrared facilities at NSLS.

Organization Chart for COMPRES DAC-XR facility at the NSLS*



*An advisory committee will be established to provide guidance and expertise.
** Endorsed by USB and BNL to the operation.

F Budget

Personnel: NSLS DAC-XR has traditionally been significantly understaffed compared to other synchrotron beamline. An increase in staffing levels is essential to realize the potential of the facility. We propose a minimum staffing level of 1 senior beamline scientist and 0.5 junior beamline scientist per station. One of the beamline scientists will be a laser-heating specialist. Increased technical support is also necessary. We propose that COMPRES support 0.5 mechanical technician for the DAC-XR facilities. As an endorsement to our plan, SBU and BNL will jointly fund a research faculty position affiliated to the DAC facilities at the NSLS to promote the scientific research program. The Mineral Physics Institute computer specialist, Ken Baldwin, will be available for computer related supports. (*The personnel supports suggested by the community at the February COMPRES workshop initially include two Beamline Scientists, two Postdocts and one technician. The requested support reflects a reduction according to the COMPRES Executive committee's advice.*)

Equipment: As the pioneer synchrotron DAC facility in the country and the world, many fundamental components and equipment need to be updated after ten or twenty years of use. The suggested major instruments to be updated or acquired by the community at the February workshop includes:

Laser heating system (update/$204K), a Mar345 detector (acquisition/$120K), 2 solid state detectors (update/$25K), a CCD detector (acquisition/$190K), DACs (acquisition/$89K) and DAC/detector tables (update/$60K), On-line ruby system (acquisition/ $20K) and laser for off-line ruby system (update/ $15K), x-ray slit system (update/$15K) and optical component (update/$39K), sample preparation (update/$32K)and gas loading systems (acquisition/ $40K), external high/low temperature system (acquisition/$46K), and Brillouin spectroscopy system (acquisition/$79K). Advised by COMPRES Executive Committee, we decide to remove the acquisition of Brillouin spectroscopy system from this five-year cycle to reduce the budget. As discussed in previous sections, the requested equipment updates/acquisitions are essential to keep the experimental capability of the facility internationally competitive. Obsolete instruments have been hindering the further advances of the techniques that were initialized at this facility.

Budgetary Summary

Budget Category	Year 1	Year 2	Year 3	Year 4*	Year 5	Total
Salaries and Wages						
Two senior/one junior beamline Scientists and half technical Associate	$191,010	$199,324	$205,725	$200,888*	$207,598	$1,004,545
Fringe Benefits	$69,719	$76,740	$83,319	$85,377*	$92,381	$407,536
Total Salary, Wages & Fringe Benefits	**$260,729**	**$276,064**	**$289,044**	**$286,265**	**$299,979**	**$1,412,081**
Supplies	$4,500	$4,635	$4,774	$4,917	$5,065	$23,891
Services	$25,000	$25,750	$26,523	$27,318	$28,138	$132,729
Travel	$10,000	$10,300	$10,609	$10,928	$11,256	$53,093
Total Direct Costs	$300,229	$316,749	$330,950	$329,428	$344,438	$1,621,794
Indirect Costs	$99,826	$105,319	$110,041	$109,535	$114,526	$539,247
Total Operation Cost	$400,055	$422,068	$440,991	$438,963	$458,964	$2,161,041
Capital Equipment	$233,000	$222,000	$215,000	$207,000	$18,000	$895,000
TOTAL	**$633,055**	**$644,068**	**$655,991**	**$645,963**	**$476,964**	**$3,056,041**

* Salaries of Year 4 reflect down grade adjustment due to anticipated retirement of current beamline scientists in previous year.

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

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

Current funding for 2002-2007: $575K

Funding requested for 2007-2012:
$1560K for operations
$490K for equipment

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.

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

A. 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.

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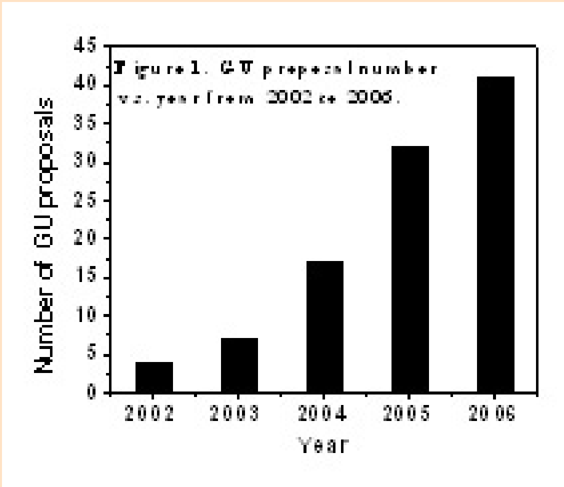

Fig 1. GU proposal numbers vs year from 2002 to 2006

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.)

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.

Fig. 2. Group Photo from the IR Workshop

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.

B. 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 and 4 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.

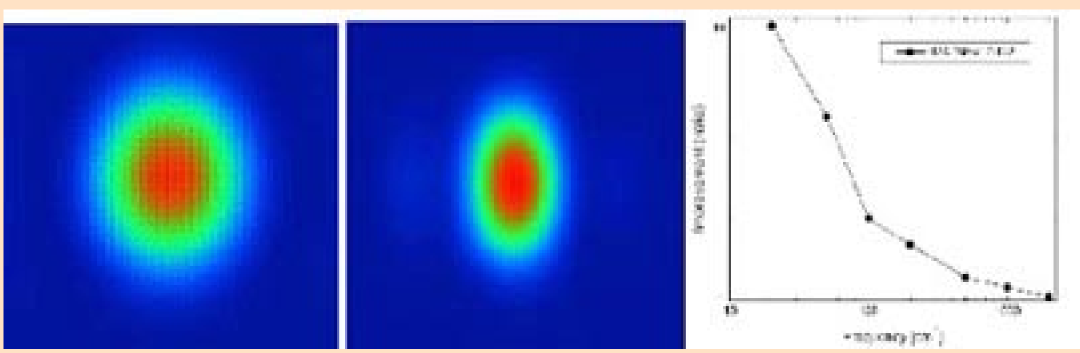

Fig 3. Diffracted beam profile for λ= 200 μm (50 cm^{-1}). Left: U2A with old 38 mm diameter optics. Center: U2A with 76 mm diameter optics. The red oval represents the profile of the collection optic at the end of the 12 m transport length. The scale is the same for both images. Right: Relative performance improvement for an upgraded U2A beamline (same length as present, but with 2× 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 H_2O and D_2O ice VIII at low temperatures up to 20 GPa and compared with the results of a series of first-principle studies for this phase transformation.

The beamline capability expanded into the new far-IR region at low-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 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. 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. Since the clathrate is more compressible than dense ice phases, a question is raised about whether it may be possible to achieve the condition where the hydrogen bonds in the cages formed by the water molecules could be driven to a symmetric state as that observed in a high pressure form of pure crystalline ice, ice VIII.

A series of far- and mid-IR measurements were carried out by Klug *et al*. to identify the predicted phase transition from non-symmetric to symmetric hydrogen bonds at both room and low temperatures up to 40 GPa. Pressure dependence of the C-H and uncoupled O-H stretching vibrational

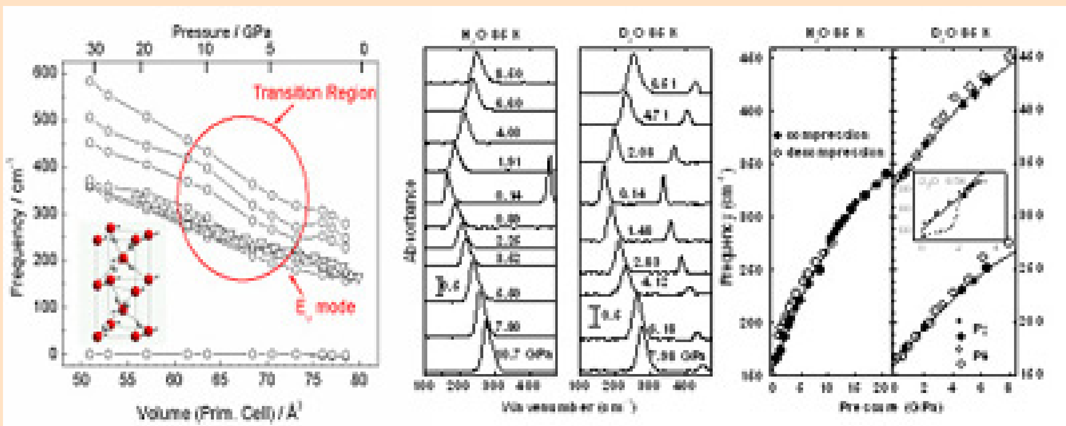

Fig 4. Right: The calculated pressure dependence of the lattice modes of ice VIII. Center: The measured low-temperature far-infrared spectra of ice VIII in a diamond anvil cell. The low frequency peak is the infrared active E_u mode. The higher frequency peak is an infrared active mode due to molecular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

modes indicates that the possible center symmetric hydrogen bonds as well as quantum phase may initiate at about 20 GPa. This finding is consistent with the theoretical density functional studies of the stability and phonons for this clathrate system. Another important feature of these experiments was the characterization of the significant temperature dependence of Fermi resonance in this system. [D. Klug, Phys. Rev. Lett, submitted].

c. 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 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)

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

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.

Single-crystal samples of LCEN with varying water contents were synthesized in a multianvil press at 16-18 GPa and 1100ºC at the Geophysical Laboratory. Samples containing ~300 and ~600 ppm H_2O were selected for the high-pressure IR study. In

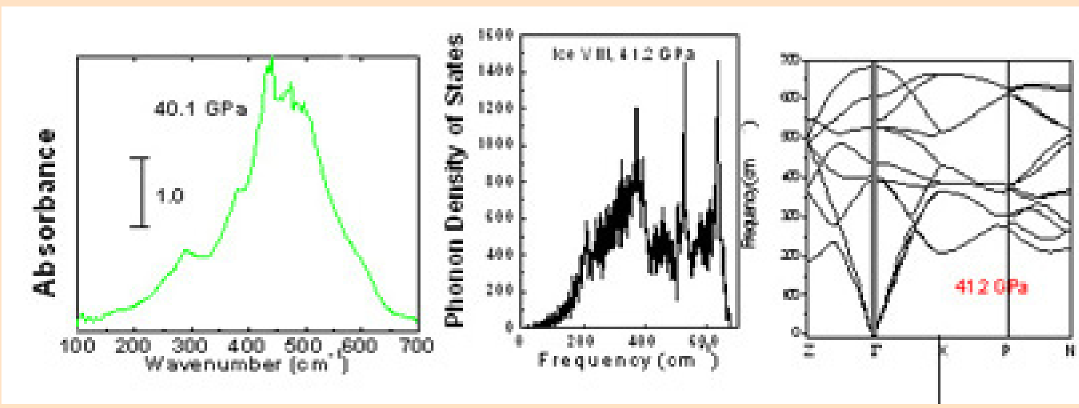

Figure 5. Synchrotron far-IR spectra of ice-VII (right), calculated phonon density of states (center), and the phonon dispersive curve at room temperature and ~40 GPa.

a. Effects of Water on the Behavior of $MgSiO_3$-Clinoenstatite at High Pressure

Just 1000 ppm (0.1 wt%) of H_2O 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 ($MgSiO_3$) 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-

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 H_2O, 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 H_2O 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 ($Mg_3Si_4O_{10}(OH)_2.nH_2O$) 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 ºC 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

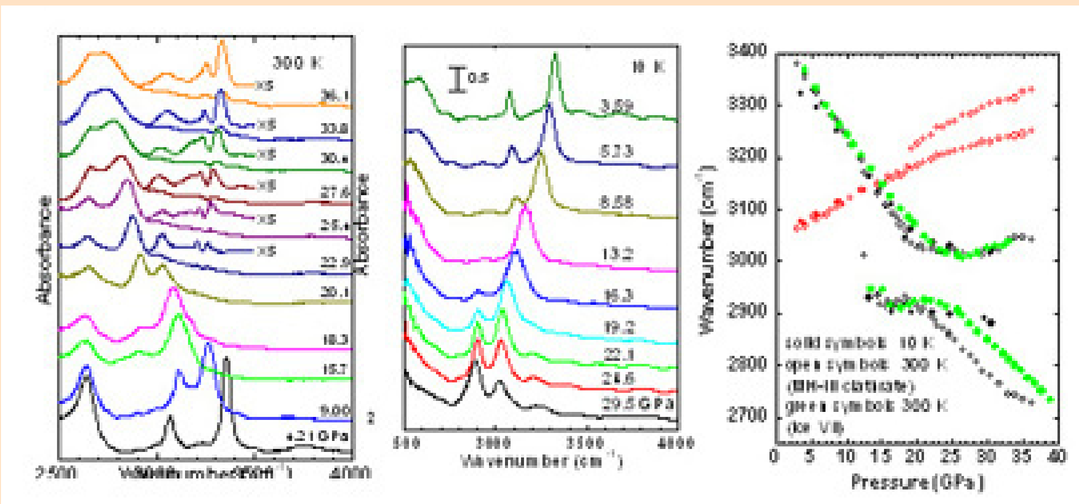

Fig 6. High-pressure synchrotron IR spectra of the CH4 clathrate at room temperature (right), 10K (center), and pressure dependence of the frequencies of OH and CH stretching vibrational modes (right).

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 SiO_4 tetrahedra, MgO_6 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 $Na(NaMg)MgSi_8O_{22}(OH)_2$ amphiboles up to 30 GPa were carried out by Gianluca Iezzi *et al*. These minerals represent a key double-chain silicate to model the $P2_1/m – C2/m$ phase-transition in A-site filled amphiboles. It has $P2_1/m$ 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 ^{A}Na; this pattern is typical of an amphibole with a *P*-lattice. The ^{B}Li-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 ^{B}Na end-member, the A and B bands merge at around 18 GPa; for sample 406, with nominal B-site composition $(Na_{0.2}Li_{0.8}Mg_{1)}$, the A and B bands merge around 13 GPa. These results show that the $Na(NaMg)MgSi_8O_{22}(OH)_2$ amphibole undergoes a $P2_1/m – C2/m$ 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

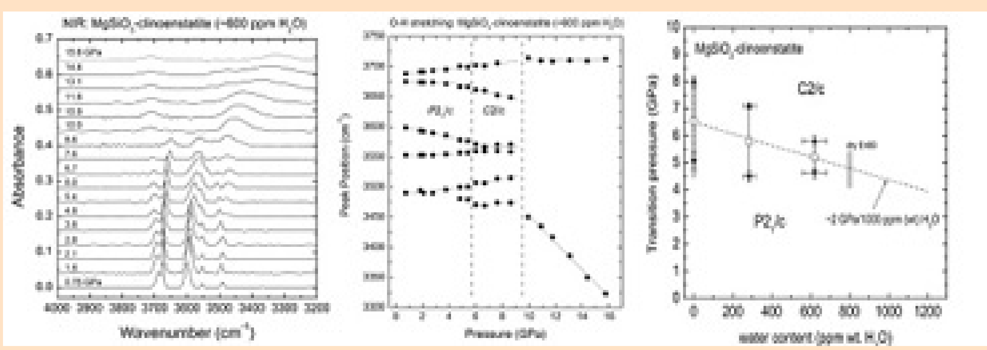

Fig 7.. Left: High-pressure IR-absorption bands due to O-H in clinopyroxene measured at U2A. Center: Variation of the O-H stretching band positions with pressure. Here we show how changes in the hydrogen bonding environment at the LCEN ($P2_1/c$) to HCEN (C2/c) phase transformation are detected using 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.

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 ($CaSO_4$•$2H_2O$) is one of the most common sulfate minerals, forming in a variety of environments including hydrothermal vents near mid-ocean ridges, diagenetically altered marine sediments, and evaporate deposits. It contains both molecular water and molecular-like sulfate groups ionically bounded to calcium ions. Recent high-pressure Raman and IR studies give different interpretations compared 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 5GPa. Both IR and ADXD showed that this high-pressure is fully reversible. It also revealed that the white synchrotron radiation induced dehydration due to the intrinsic features of this high-pressure phase in gypsum during the EDXD measurements. This quenchable high-pressure phase has been studied by IR and Raman as well and is determined as following:

$CaSO_4$•$2H_2O$→high pressure phase (orthorhombic ~5GPa)→ $CaSO_4$•$0.8H_2O$.

[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, $Ca_3Al_2(O_4D_4)_3$ up to 10 GPa by infrared absorption. Discontinuities in both O-H and O-D vibrational frequencies at ~5 GPa suggest a pressure-induced phase transition that is in excellent agreement with the single crystal x-ray diffraction studies at high pressure. Pressure dependence of the frequencies related to the O-H stretching vibrational modes is consistent with the calculated results and indicates that deuteration does not significantly affect the pressure of the transition. [Am. Mineral., **90**, 639 (2005)].

II. Scientific and Technical Challenges for the Near Future and a Vision for NSLS-II

A. *High P-T synchrotron IR studies of hydrated transition zone and lower-mantle minerals*

It is possible that the majority of the planet's H_2O budget resides deep in the interior as structurally bound hydroxyl (OH) in high-pressure phases. The ability of deep-mantle minerals to store water (hydrogen) depends on many factors such as bulk composition and temperature, but structural factors dominate. Whereas the olivine structure down to 410 km-depth can only store up several thousand ppm H_2O by weight [*Chen et al., Geophysical Research Letters,* **29**, doi: 10.1029/2001GL014429, 2002], both wadsleyite and ringwoodite in the transition zone (410-660 km depth) can contain tens of thousands of ppm H_2O [*e.g. Jacobsen et al., American Mineralogist,* **90**, 2005]. In the lower mantle, where silicate perovskite (PV) and magnesiowüstite (MW) are the stable assemblage, the water storage capacity remains uncertain, with current estimates ranging by three orders of magnitude, from ~1 ppm [*Bolfan-Casanova et al., Geophysical Research Letters,* **30**, doi: 10.1029/2003GL017182, 2003] to over 2000 ppm wt. H_2O [*Murakami et al., Science,* **295**, 1885, 2002] in silicate perovskite. The reported range of water contents in perovskite depend largely on how the SIMS (ion probe) or infrared spectra are interpreted. Although ion probe measurements detect H in synthetic Si-perovskites grown under hydrous conditions, only IR-spectroscopy affords the opportunity to provide information on the speciation of hydrous components. The major question now is whether or not hydrogen observed in synthetic samples occurs as structurally bound hydroxyl in the perovskite, or is present as hydrous mineral inclusions or melt quench. To date, all studies on the incorporation of water into Si-perovskite have relied on samples synthesized from mixed oxides and measured at ambient conditions after quenching from high *P-T*. Recently, S. Jacobsen et al. have directly transformed hydrous ringwoodite to PV+MW in a laser-heated diamond cell (Fig. 9) at the GSECARS beamline of APS and studied IR-absorption in the O-H stretching region at synthesis pressures in-situ at U2A beamline

These ongoing studies have pointed toward exciting new research directions on all deep- and lower mantle hydrous minerals using synchrotron IR technique combined with high P-T DAC techniques. However, it is necessary to have an onsite laser heating system opened to all users. We therefore propose to develop high pressure and temperature infrared spectroscopy beyond the resistive heating barrier (1200 K and higher) at U2A beamline. The first step is to build an offline CO_2 laser heating system with standard temperature calibration technique. Further development will focus on new temperature calibration method, such as collecting black body thermal radiation using small FTIR spectrometer to cover the 800~1500 K gap; coupling the high spatial resolution of the synchrotron source with the CO_2 laser technique for *in situ* high P-T mid-IR experiments.

B. *Diffraction limited imaging technique, THz spectroscopy, and side station*

The U2A beamline has been built as the first dedicated high pressure IR beamline in the world with many unique features, such as vacuum far-IR microscopic system for measurements down to the THz region (1 THz=33 cm^{-1}), and integrated synchrotron IR/Raman/visible spectroscopy at high pressure and variable temperature (4-1000 K). Despite these features, there are important limitations in the present configuration that preclude optimized performance. These limitations are becoming particularly important as the IR user community grows. The existing Bruker spectrometer and microscope is not optimized for IR imaging which is particularly important for geological specimen. The distance between the U2A beamline end station (spectrometer/microscope) and the synchrotron source spot is about 15 meters, 3-5 times longer than the other five IR beamlines at NSLS. The synchrotron beam is collimated with a parabolic mirror and delivered with several flat mirrors through a vacuum pipe system; we have found that the beam divergence, scattering, and distortion becomes significant after the so-called "collimated" beam travels more than 8 meters. As a result, the performance at U2A is significantly lower (by a factor of two) than the other beamlines in the mid-IR. This limitation poses problems for experiments that require the highest spatial resolution (e.g., IR mapping of samples down below 5 µm or the diffraction limit). There is increasing interest from the high-pressure geoscience community in conducting these kinds of experiments.

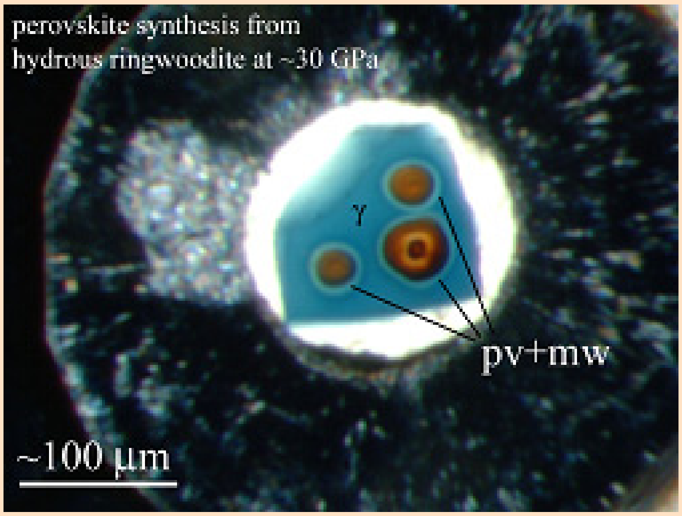

Fig 8. Single-crystal hydrous ringwoodite (blue) laser heated to PV+MW at several temperatures (1500, 1700, and 2000 K: orange spots). The distribution of water in the sample was studied using synchrotron-IR at U2A at synthesis pressures.

During the past year, an exciting opportunity has arisen to create a side station on the beamline as a result of new space that has been created next to the U2 port. The distance from the synchrotron source to the IR system would then be only about 3 meters. This will remove the problem of beam divergence and image distortion. Ray-tracing calculations have indicated a significant improvement in spatial resolution (equivalent to U10B, which is essentially identical to the proposed side station). The new facility allows measurements on high-pressure samples with the highest spatial resolution possible at a synchrotron source while also having the highest broadband IR brightness. With a new microscope coupling a newly developed IR CCD detector and FTIR instrument, the facility will be ideal for mapping of natural samples (e.g., solid and fluid inclusions in thin section), heterogeneous charges from high-pressure experiments, as well as samples *in situ* at very high pressure in diamond or moissanite anvil cells. As such, the side station facility would therefore

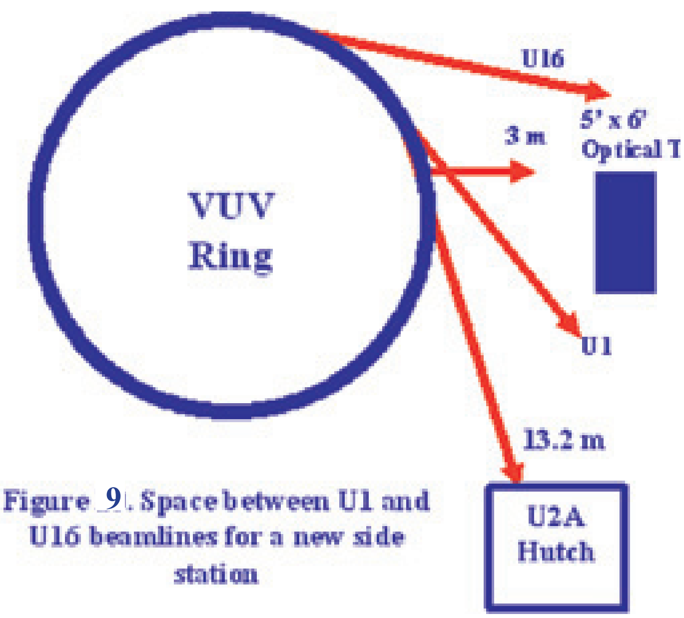

Figure 9. Space between U1 and U16 beamlines for a new side station

complement the main beamline, with its general purpose microscopes, vacuum capability, far-IR features, and laser spectroscopy systems. Optics will be available to split the beam and allow us the option of operating both the existing and the new station alternately. In addition, this side station will provide new feasibility for multidisciplinary high pressure science beyond Earth Science applications, such as nano-scale materials and explosive materials.

C. <u>Vision for the NSLS-II</u>

The NSLS-II project, a proposed replacement to the existing NSLS x-ray ring, includes a significant infrared component. Like the existing NSLS, the NSLS-II project plans call for a large 3rd generation storage ring optimized for ultra-high brightness VUV and x-ray beams plus a smaller, low energy ring optimized for infrared measurements. This low energy IR Ring will be based on the existing VUV/IR ring, but with suitable improvements and upgrades. For example, by operating the ring at somewhat lower beam energy, higher average beam currents can be supported. Combined with top-off injection to overcome the poorer lifetime, a doubling of the average beam current (over the existing ring) is anticipated. An upgrade of the RF system to 500 MHz (possibly higher) will produce shorter electron bunches. Special operating modes to produce particularly short bunches (10 ps or less) will be useful for time-resolved spectroscopy, plus allow for a stable coherent synchrotron radiation mode for strongly enhanced THz radiation as shown in Fig. 10.

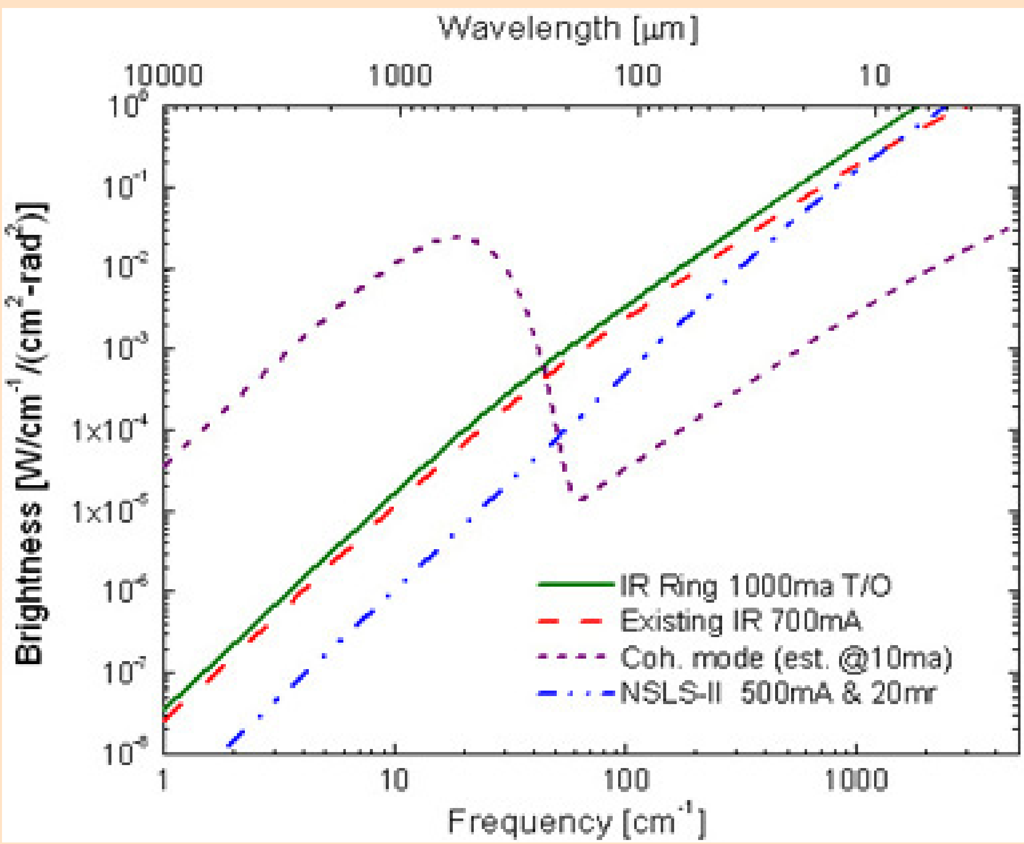

Fig 10. Calculated brightness for IR Ring (at 1a) in the NSLS-II project in comparison to the existing VUV/IR ring at 700ma and the main NSLS-II ring (for x-rays). Also shown is a conservative estimate for the THz enhancement due to the CSR from short electron bunches.

Though the specific siting for the IR Ring in the NSLS-II project has not been determined, Fig. 12 shows a candidate layout with the ring built below grade to facilitate top-off injection. Beam pipes will bring the IR light up to the ground level where ample space is available for beamline instrumentation. About 8 beamlines are expected during the initial phase of operations, including ports and end stations for high pressure and high temperature experiments. Therefore, all developments proposed above will be well accommodated into the state-of-the-art facility.

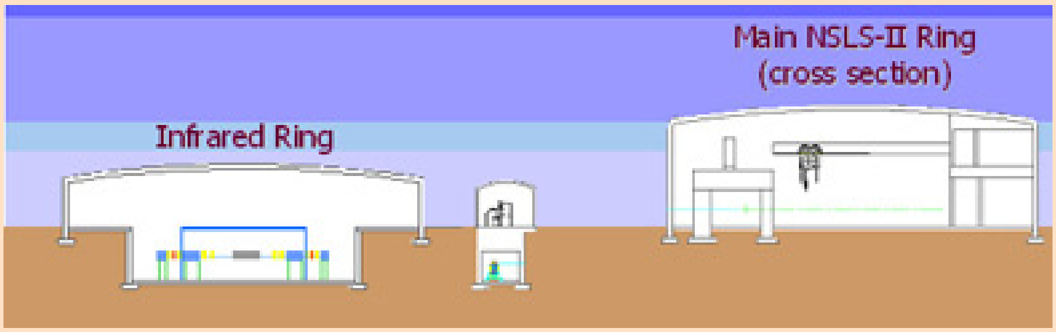

Figure 10. Schematic view of a siting scenario for the IR Ring. The ring sits below grade (same as the linac injector, shown in the center) with beamlines and endstations on the adjacent ground level floor.

III Budget Requests for the DAC-IR Operations and development in the next COMPRES grant

As discussed above, tremendous progress has been made during the past four years. However, the DAC-IR facility was seriously underfunded in the beginning. Actually, it has the lowest funding level in terms of available beam time for users/staff compared to other COMPRES supported facilities (>200 days beam time of which 50% 0allocated to general users, with only one beamline scientist to take care of users, develop new techniques, and carry out research). With a growing user community, it will be difficult to sustain the high quality of current operations, and impossible to do any new development as mentioned above without increasing manpower. Therefore, we request at least one more full-time staff position at the postdoctoral research associate level. The beamline staff will be responsible for serving users, performing his/her research, and assisting the PIs in beamline new development. An additional technician or software engineer shared by other user facilities at the NSLS (X17B2, B3, and C) will be very helpful as well.

It should be pointed out that all major equipment at the U2A beamline is close to or older than ten years. Several equipment malfunctions occurred in the last year and caused delayed experiments for general users. The proposed side station is crucial to assuring the availability of an alternative setup for users. The detailed budget request is shown in the following table; this funding level will assure us the ability to serve the user community as well as to keep the DAC-IR beamline operating as a world-class facility.

Total proposal budget over 5-year period of COMPRES II

Budget Category	Year 1	Year 2	Year 3	Year 4	Year 5	Total
Salaries/Wages	$135,000	$141,000	$147,000	$153,000	$160,000	$736,000
Fringe Benefits (28.5%)	$38,475	$40,185	$41,895	$43,605	$45,600	$209,760
Travel	$9,000	$9,500	$10,000	$10,500	$11,000	$50,000
Materials/Supplies	$41,335	$45,029	$48,724	$52,419	$54,829	$242,336
Total Direct Cost	$223,810	$235,714	$247,619	$259,524	$271,429	$1,238,096
Indirect Cost (26%)	$58,190	$61,286	$64,381	$67,476	$70,571	$321,904
Total D&I Cost	$282,000	$297,000	$312,000	$327,000	$342,000	$1,560,000
Equipment	$80,000	$230,000	$100,000	$80,000	$0	$490,000
Total Requested	$362,000	$527,000	$412,000	$407,000	$342,000	$2,050,000

Budget justification for the first year (May 2007-April 2008)

With a significant increase in the amount of beam time available to general users and contributing users as well as plans for beamline upgrade, we request $362 K for operating, maintaining, and upgrading the IR beamline for the year beginning May 1, 2007 and ending April 30, 2008. For justification, the total expense is shown as following:

Budget for Beamline operation

Personal: Beamline scientist Postdoctoral associate		Salary plus fringe benefit (28.5%)	**$102.8 K** **$70,675 K**	This includes a normal raise and cost of living adjustment.
Materials/supplies				
	a.	Liquid helium ($450/60L, 22 tanks in total)	$9.9 K	This reflects the increase use of the FIR system where it is necessary to cool the Si bolometer and high-P and low-T experiments.
	b.	Stockroom& NSLS fees	$12 K	This reflects routine beamline expense including offices, phone, and supplies.
	c.	Diamond anvils (6)	$9 K	Low fluorescence type Ia and IIa diamond anvils are required for dedicated use at the facility.
	d.	Optical components	$10.435 K	ThorLab optical components to be used to set up an enclosed microscope system. This will allow users to calibration pressure without any laser safety requirements as the enclosed laser can be treated as class-I laser.
		Sub-total	**$41.335 K**	
Travel		Travel for beamline scientist, post-doc and spokesperson	**$9 K**	This is needed for travel between BNL and Washington as well as for the beamline scientist and post-doc to attend COMPRES and scientific meetings.
		Total direct cost	**$223.81 K**	
Total cost for operation			**$282 K**	This reflects the Carnegie off campus overhead rate. ($223.81K*1.26)

Budget for permanent equipment

Capital equipment (CO_2 laser heating system)	*SynRad Firestar* 200W Sealed CO2 Laser	**$33 K**	High power CO_2 laser for generating high temperature in DAC under high pressure.
	Spectrograph with gratings	**$5.5 K**	PI Acton SpectraPro SP-2156 Imaging Spectrograph, 150mm focal length.
	CCD detector with shutter & software	**$20 K**	The spectrometer and CCD detector will be used to measure the thermal radiation curve and determine the temperature
	Optical components	**$21.5 K**	Parts will be used to build a custom microscope system with viewing/ focusing functions
Total request for equipment		**$80 K**	
Total Request Budget		**$362 K**	

Budget justification of the capital equipment for the years 2008-2010

In order to keep the world-class IR facility at the NSLS, we will make several critical beamline upgrades to accommodate user's need. As shown in the budget justification for the first year, we request $80 K for a CO_2 laser heating system. In addition, we request $410 K to purchase permanent equipment for years #2, #3, and #4 in order to develop new techniques and capabilities for users from the community. For justification, the total expense is shown as following:

Bruker Bruker	Vertex 80 FTIR spectrometer with accessories Focal Plane Array Detector	**$145 K** **$85 K**	This will be next major upgrade after the CO_2 laser-heating project. These are the core equipment for the side station discussed in the section IIb. The construction of a vacuum pipe system for synchrotron beam delivery is on the way and will be completed in this year. This side station is also critical to combined the CO_2 laser heating technique and make in situ high PT capability available for users.
Coherent	Ar ion tube and solid state green&blue lasers	**$50 K**	U2A beamline provide an access for all users to do not only synchrotron FTIR spectroscopy under extreme conditions but also Raman scattering. The Ar ion laser is more than ten years old and the tube (~$20 K) need to be replaced. In addition, we need diode pumped solid-state green&blue lasers for the side station (~$30 K).
Roper Scientific	Online ruby pressure calibration system	**$40 K**	This system with spectrograph and CCD detector will be combined with the FTIR spectrometer at the side station for pressure calibration.
Leica Hylozoic Products	Microscope EDM machine	**$15 K** **$0.7 K**	The Leica microscope, EDM machine and sample loading system will form a sample preparation center for users.
Home made	Cryogenic sample loading system	**$0.5 K**	
Cryo Industries of America, Inc	Cryostat	**$35 K**	New development is needed to do better low-T experiments.
Home made & Diacell Products Ltd.	Diamond anvil cells and accessories	**$28 K**	A serious diamond anvil cells are needed for different experiments at variable temperature and pressure.
Total request		**$410 K**	

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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
Funding requested for 2007-1012: $2176 for operations
$ 862K for equipment

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.

We propose to continue to operate X17B2 at the NSLS for large-volume, high-pressure research for the next five years for the COMPRES community. Donald Weidner will serve as PI.

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 2002-2006

- ***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, $Mg_{0.9}Fe_{0.1}SiO_3$, 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-quenchable 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 $MgSiO_3$ perovskite, Al-doped $MgSiO_3$ perovskite, non-quenchable $CaSiO_3$ 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 cure. 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.

Science Objectives for 2007 - 2012

Phase transitions

Experimental and theoretical investigations of phase transitions in Earth and planetary materials under the pressure and temperature conditions of their interiors are of fundamental importance to our understanding of the nature and dynamics of planetary bodies in the solar system. In the next five years, data on phase transitions in several areas have been identified as of primary interest to the study of the Earth and planetary interiors. For crustal and upper mantle materials, new experimental data on the phase transitions in ultra-high-pressure metamorphic rocks are needed to assist the study of deep focus earthquakes. A thorough investigation of the post-spinel transition will help interpret detailed seismic observations on the transition zone, including the magnitude, sharpness, lateral heterogeneity, and topography of the 410 and 660-km discontinuities. Further studies of the post-perovskite transition, including the determination of the Clapeyron slope, are necessary for unraveling the mysteries concerning the D” zone and ultra-low-velocity-zone at the core-mantle boundary. A new class of phase transition in planetary materials such as CAIs, chondules, and basaltic glasses from the Moon promise to shed light on the origin and evolution of the early Earth.

Factors such as stress, multi-components, grain size, need to be quantified as we relate laboratory results to the Earth. Before equilibrium is reached, phase transitions proceed as a function of time. Investigating the kinetics of phase transitions is important for assessing the fate of the subducted slabs and understanding the dynamics of the core-mantle boundary.

Melt and glass structure

High resolution x-ray diffraction for crystallographic studies, and high energy x-ray scattering for pair distribution function (PDF) studies of non-crystalline materials (melts and glasses) at high pressure pressure/temperature has drawn great attention among the participants at the workshop. While properties of crystalline minerals have been extensively studied from the crystallographic point of view, melts and glasses increasingly become of geophysical interest because melts and partial melting play an important role in mantle dynamics.

Structure determination of glasses and melts at high pressure and temperature require high quality data. These data must be free from parasitic scattering from cell components, such as: anvils, gaskets, and furnace materials. Several methods are used to eliminate or reduce background scattering and are applied either in the data processing stages or preferably during data collection. These include: background measurement and subsequent subtraction from high PT data and/or the use of slit systems to completely eliminate parasitic scattering from reaching the detector. In practice, it is generally not enough to simply collect backgrounds at ambient conditions and apply these to high PT data because backgrounds change with P and T, and may not be easily applicable. On the other hand, Soller slit systems, such as the one illustrated in Figure 1, can be used to completely eliminate parasitic scattering from reaching the detector and are functional at any PT conditions. Soller slits, when properly installed and aligned, can provide high quality data.

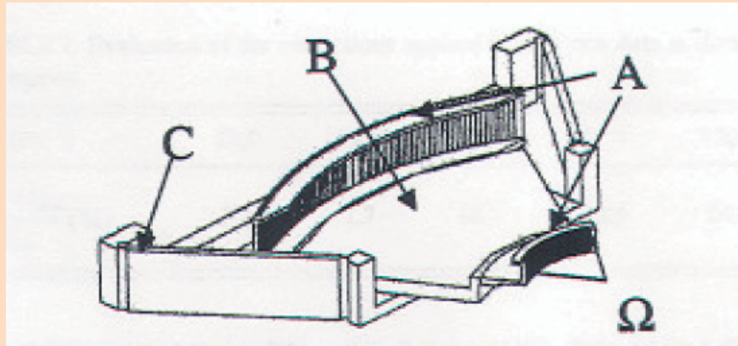

Figure 1. Illustration of a Soller slit system (multi channel collimator) used at the ESRF. (A) inner and outer Ta slits, (B) mounting plate, and (C) a rotator. Figure is taken from Mezouar et al., 2002 [2].

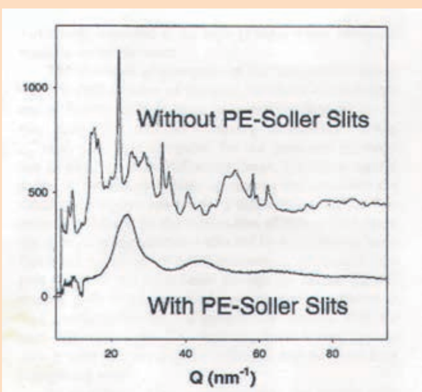

Figure 2. Diffraction pattern of liquid tin with and without Soller slits.

Soller slits are currently being used with multi-anvil (Yaoita *et al*, 1997) and Paris-Edinburgh (Mezouar *et al.*, 2002) pressure systems. An example of liquid diffraction data collected using a Paris-Edinburgh press, with and without Soller slits, is displayed in Figure 2. This figure illustrates a very important point about liquid (melt) and glass scattering: namely, melts and glasses are very weak scatters and their signals can be washed out by parasitic scattering. However, it is immediately apparent that Soller slits are remarkable at removing parasitic scattering and allowing the collection of the high quality diffraction data necessary for structure determination of glasses and melts at high PT conditions.

Elasticity

The most robust fingerprint of chemical and thermal state of the Earth's interior is the set of elastic properties Earth materials. Radial variations in seismic velocity point to phase transitions, melting, and general pressure increase. These transitions require a comprehensive understanding of the elastic properties of materials as a function of all of the relevant variables. The last few years has seen tremendous growth in our data base as well as our experimental tools for defining this information.

The interpretation of seismological profiles of Earth's interior has long been the principal motivation for measuring the acoustic velocities and the elastic tensors of minerals, both at ambient and high P or T conditions. As the resolution of seismological studies continues to improve, the need for more and better elasticity data, under simultaneous high pressures and high temperatures, increases.

Two specific challenges that can be highlighted include: the interpretation of seismic anisotropy throughout the planet, from uppermost mantle to inner core conditions; and understanding lateral variations of compressional and shear wave velocities (∂Vp and ∂Vs) in terms of composition and/or temperature variations. These goals require the mineral physics community to provide complete characterization of elastic anisotropy, as well as aggregate acoustic velocities, in minerals, and also the variation of these properties with pressure, temperature, and composition.

Simultaneous ultrasonics+XRD investigations in the multi-anvil press permit the EoS and acoustic properties of minerals to be evaluated under high-P,T conditions. An example is the recent study of $MgSiO_3$ perovskite to 9 GPa and 873 K by Li and Zhang (2005). In principle this technique can be extended to 20 GPa and 2000K.

Finally, it should be noted that with synchrotron XRD techniques, characterizing stress and strain is now done routinely in a variety of high-P,T experiments. This is an important advance over past experimental methods, in which the state stress/strain of the samples were commonly uncertain or unknown.

In order to further develop the ultrasonic studies at X17B2, a 2000-ton press with exchangeable modules is immediately needed to expand pressure ranges of a variety of experiments, including equation of state, phase transformation, and ultrasonics. Sintered diamond cubes are needed for expanding the pressure to the top of the lower mantle pressures to narrow gap/expand the overlap in pressure range between diamond anvil and large volume apparatus to ensure consistency from different techniques. These new acquisitions will benefit the experiments for equation of state, ultrasonics, and lattice strain studies.

Rheology

The quantitative relationship between stress, strain, and time in minerals forms the basis for our view of the evolving Earth. Plate tectonics, earthquakes, volcanic eruptions all respond to these intrinsic properties of Earth materials. Laboratory studies have recently made a significant breakthrough in capability for defining these properties at mantle pressures and temperatures using x-rays generated by synchrotrons at national laboratories. This progress has set the stage for new and exciting research efforts.

Significance of deviatoric stress measurements at high pressure. Thermal convection in Earth's deep interior cools the planet and in the process generates earthquakes and volcanoes, moves tectonic plates, and disturbs the uniform chemical layering of a differentiated Earth. Laboratory measurements of the relationship between deviatoric stress and deviatoric strain rate of rocks and minerals at high pressure are driven by the need to understand this circulation at depth. Characterizing the state of deviatoric stress during experiments under high confining pressure is also critical in a number of other mineral physics studies that have important bearing on the frontiers of solid Earth science (e.g., accurate characterization of seismic velocities of high-pressure phases). Current research on global geodynamics strongly suggests that the dynamics and evolution of this planet are controlled largely by materials properties under deep Earth conditions, including rheological properties, phase relationships, elastic properties and chemical properties such as the diffusivity and solubility of certain elements [e.g., [*Bercovici and Karato*, 2003; *Kellogg, et al.*, 1999; *Schubert, et al.*, 2001; *Tackley*, 2000; *van Keken, et al.*, 2002]. For instance, the lateral and radial variation of viscosity have an important influence on the convection pattern and generation of deep earthquakes [e.g., [*Bunge, et al.*, 1996; *Christensen*, 1984; *Green and Houston*, 1995; *Green and Marone*, 2002; *Karato, et al.*, 2001; *van Keken and Ballantine*, 1998], whereas the solubility and diffusivity of elements in various phases control the chemical evolution associated with mantle convection [e.g., [*Hart*, 1988; *Hofmann*, 1997; *Van Orman, et al.*, 2002]. Also, the way in which materials are distributed or the flow pattern in Earth can, in principle, be inferred from seismological observations, but the interpretation of seismological data relies entirely on our understanding of elastic and anelastic properties of minerals under deep Earth conditions [e.g., [*Jackson*, 2000; *Karato and Karki*, 2001; *Karki, et al.*, 2001; *Liebermann*, 2000].

In the experimental study of these physical properties, characterization of stress plays an important role in one way or another. For example, in the study of rheological properties, the relation between (deviatoric) stress and strain rate is most appropriately determined under the conditions equivalent to those in Earth's interior. Consequently, precise measurements of stress and strain at high pressure have direct influence on the quality of the experimental results. In particular, the demand for high-resolution stress measurements is critical for the study of rheological properties in order to obtain results that can be extrapolated to Earth's interior. In addition, some transport properties, such as diffusion, should be measured under low deviatoric stress because dislocations generated by deviatoric stress are known to have a strong influence on diffusion coefficients [e.g., [*Flynn*, 1972; *Shewmon*, 1989]. Defects such as dislocations and grain-boundaries may also have an important effect on element partitioning [e.g., [*Hiraga, et al.*, 2004].

Recent progress. During the last few years, there has been important progress in measuring the state of stress of samples during high-pressure experiments. Firstly, there have been significant advances in high-pressure, high-temperature deformation apparatuses, in particular, the successful development of two instruments: the Deformation DIA (D-DIA) [*Wang, et al.*, 2003], and the Rotational Drickamer apparatus (RDA) [*Xu, et al.*, 2004; *Yamazaki and Karato*, 2001], by which quantitative rheological experiments have become feasible well beyond a pressure of ~4 GPa. Insights provided by these new instruments will be key to better understanding of the evolution and dynamics of terrestrial planets. An important new dimension currently in its early stages is development of ways to use the diamond anvil cell (DAC) to extend such deformation studies to much higher pressures.

We have recently developed a method for measuring stress in a sample under high-pressure and temperature conditions in a multianvil apparatus using x-ray diffraction from a synchrotron source *[Li et al, 2004]*. The spacings of the lattice planes are measured both parallel and perpendicular to the principal stress axes. The stress is then derived from this measured elastic strain using the elastic moduli [*Singh, et al.*, 1998]. We have used both white x-ray and monochromatic techniques. For the white beam studies, we have used a multi-element solid-state detector that was designed for EXAFS studies. With it we use four elements that are positioned at 90^{o} from each other. A conical slit system was designed and built to fit the detector. Because of the small diameter of detector, we could not build a slit system that optimized the optics such as the acceptance angle and spatial resolution. Nevertheless, we obtain precision of 100 MPa. The number of detectors in our current system precludes defining the orientation of the principal stress axes projected on the plane of the detectors (so they must be known *a priori*), and the dimensions of the slits limits the x-ray resolution. The monochromatic system yields about the same precision, with a greater ability to define the axis of the stress field because a 2-D detector is used. However, the monochromatic system cannot readily collimate the diffracted x-ray beam, so the background due to diffraction from the pressure medium and parts of the sample assembly can easily hide diffraction from the sample, which limits our choices of building materials.

At the workshop the following issues were expressed:

- In order to characterize the flow properties of many important earth phases, we need higher pressure. Although the DAC can provide much wider pressure range than LVP, there are still concerns about whether the DAC is an appropriate tool for the characterization of flow laws.
- Quantify effect of H_2O on rheology; Progress has been made but there is more work to be done especially at high pressure. We do not fully understand where water resides – in

crystals? Along grain boundaries? Does water stay in the same place upon quench? Do we need to look for in-situ techniques that will allow us to probe the structural sites for water at high pressure and temperature? Or are quenched samples representative?

- Improve stress resolution to 10 MPa. Making good measurements at low stress levels will be important for properly characterizing activation volumes
- Confirm that we measure the flow mechanisms appropriate to the mantle
- We need to better understand the effect of other volatiles and impurities on rheology.
- We need to better understand the character of grain boundaries at high pressure and temperature as they are likely to become more like defects (or grain boundaries in metals). If atoms on the grain boundary behave as if they are in the liquid state then we can anticipate a larger pressure effect on their behavior than we might otherwise anticipate.
- The small probe size of NSLS II opens up the possibility of nano-imaging or tomography techniques that will allow us to observe defects in-situ during deformation.
- Techniques that allow us to create high strains will be important for studying the development and effects of lattice preferred orientation and the development and effect of rock textures (e.g. two phase mixtures, foliated or textured rocks etc.)

Challenges:

- **Strain measurement** of 10^{-6} would allow us to do anelasticity measurements with small source size. At NSLS II this would be possible. We will need to build optics to enlarge the image before we convert it to light (x-ray microscope)
- This higher resolution would also improve deformation measurements in the DAC (their sample size is small which limits their ability to measure strain.)
- Possibly single crystal Q experiments combined with polycrystalline Q experiments would allow us to examine “micro-creep” vs “macro-creep” and therefore shed light on the interpretation of post glacial rebound data.
- Develop cells that can go to higher temperatures than we currently achieve.
- Software **data processing** issues: need to get high precision stress/strain results in real time.

Current status at X17B2. We now have a design for the conical slit system with a new detector that is optimized for stress resolution. We expect that we can achieve 10 MPa precision with the new system. We have also recently purchased (with a grant from the Air Force) a MAR345 imaging plate detector which will be used with monochromatic x-rays. We will be able to refine stress measurements with this system. We are nearing the final design of a D-Tcup which is a deformation system similar to the DDIA, but using the T-cup tooling. We expect that this system will allow us to carry out deformation experiments at high temperature and pressures exceeding 20 GPa. We currently have funds for the purchase of this system.

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

Large Volume High Pressure Budget

Operations Budget: We include in the operations budget funds for two beamline scientists, one technical support person, and funds for supplies. This represents an increase of one beamline scientist from the program of the last five years.

The NSLS high pressure workshop identified as a top priority the need to fully reduce all data during the experiment. Both rheology and elastic velocity experiments attain complex data. In the case of rheology, diffraction data for several diffraction vectors are gathered to define the elastic strain of the sample. We currently use four detectors for gathering this data simultaneously, but plan to increase this to 16. The sample stress is deduced from these data. The scientist needs to change strain rate, temperature, and pressure during the experiment. In order to make the scientifically best decisions for these changes, it is necessary to know the state of stress in the sample which comes only from a complete analysis of the diffraction data. We have recently developed software that addresses this issue for the four detector studies. However, they are not yet user friendly, and therefore, are not used in real time data analysis. An additional beamline scientist is needed to continue the development of software that will grow with changes in the detector system, to work with users to reduce the data in real time, and to train returning users to be able to use the software during subsequent visits.

In a similar manner, the ultrasonic experiments require the reduction of travel time data in order to define sound velocity. Real time analysis will enable the scientist to isolate regions in P – T space where interesting phenomena are occurring and to concentrate on these regions during the experiment.

Our goal in adding a second beamline scientist is to change from the situation where the user takes home a large number of diffraction data to one where the user leaves with physical measurements such as stress and strain or elastic sound velocities. This will mean that the experiments are better guided and more efficient than now.

Indeed, the beamtime operations have grown considerably since the beginning of COMPRES, where we operated part-time in a shared hutch. Now, while we receive beam for ½ of the time, in the other half, we have access to our hutch. During this time, new items are built and installed. Off-line experiments can be done. Furthermore, with the side station coming on line, we expect to operate two systems simultaneously with different users on each system. This, with the anticipation of further growth of beamtime as X17A comes on line underscores the need for a second beamline scientist just for operations.

We include funds for a full time technical support person. As indicated in the table below, we actually receive more technical support than a single person from Stony Brook University. While we do not have written commitments, we anticipate that this support will continue.

We include $40,000 for supplies. Users that are given beamtime by NSLS or COMPRES have passed through a careful review of their proposed study. We feel that they need to only bring their sample to accomplish their experiment. We provide cell assemblies and anvils unless they have special needs, in which case we may request that they furnish some of these items. Many of the experiments require sintered diamond anvils or cubic boron nitride anvils. These anvils are over $1300 each and are often broken. We also furnish standard cell assemblies. This system is much more efficient than having the user furnish these items. We can maintain quality control and explore various sources of these items.

In this budget, we outline several initiatives that will increase the experimental program and allow us to push the science forward. In the first year, we anticipate that NSLS will develop a new beamline, X17A. The superconducting wiggler that fuels X17 produces a wide fan of x-rays. This fan is divided into three sectors, X17A, X17B, and X17C. The B and C sector have been fully developed with material science and high pressure sharing the B line (roughly 50/50 in time) and the diamond anvil cell facility at C full time. The A line has never been developed, but can be. NSLS has place the development of this beamline in the early portion of a five year plan as a monochromatic beamline. The cost of this development is well over a million dollars; no funds are requested at this time in the COMPRES budget for the implementation of X17A. The gain for high pressure is two fold. First, most of the material science users that now use X17B1 will move to X17A. This will allow the high pressure usage to grow to at least 80% of the time. This means that each of the three simultaneous high pressure installations (2 in X17B2 and DAC in X17B3) will each grow by 30% of the beam time. This gain is almost equivalent to a full-time additional beamline.

Second, we will install the high energy, low background diffraction system with a Paris – Edinburgh cell on this beamline (X17A). Thus, the high-pressure COMPRES community will gain additional time using X17A directly.

High energy/resolution studies: High resolution x-ray diffraction for crystallographic studies and high energy x-ray scattering for pair distribution function (PDF) studies of non-crystalline materials (melts and glasses) at high pressure pressure/temperature has drawn great attention among the participants at the workshop. While properties of crystalline minerals have been extensively studied from the crystallographic point of view, melts and glasses increasingly become of geophysical interest because melts and partial melting play an important role in mantle dynamics. As high photon energy is a unique feature of the superconductor wiggler of X17, development in this direction is suggested with a high priority. We plan to install a Soller slit system at the X17B2 side station, which has been proved very effective for collimating the diffracted x-rays to get clean (sample only) diffraction patterns using two dimensional area detectors at ESRF and SPring8. This is particularly important when collecting scattering data for deriving pair distribution function because the background between Bragg peaks in the diffraction pattern heavily affects the result of PDF. In addition, as a reliable derivation of the pair distribution function normally requires a large range of Q (diffraction vector) up to 30 (Å-1), a large diffraction angle (2□) becomes essential. Using a 100keV monochromatic beam, diffracted x-ray needs to be recorded at an angle of 35^o; and a 70keV beam at 50^o. Therefore, a Paris-Edinburgh cell with a torroidal cell which has a 360^o opening for x-ray diffraction is going to be acquired.

The slits used at the ESRF were produced by a company called Usinage et Nouvelles Technologies in Morbier, France. They used Ta metal as slit material. The cost for the slit system depends highly on the cost of Ta. The full cost of the slits, mounts, motor, and controllers is estimated to be around $85,000 to $100,000.

Large capacity press: The workshop identified a very significant need for a large capacity press with appropriate tooling. The optimal press size was identified as 2000 ton force capacity. This is twice the capacity of the GSECARS press and offers a wide range of possibilities for scientific studies. The large presses at SPring8 have defined the high-pressure limits of the 6-8 system, reaching 50 - 60 GPa, but needing the full range of their 1500 ton press. This system employs sintered diamond anvils to reach this pressure. We feel that it is important for at least one large volume synchrotron facility in the US to have the capability to compete with the Japanese efforts. We do not currently have a press, on a beamline, with as large of a force capacity as the SPring8 presses (the Japanese have two such presses at SPring8).

The tooling for a large capacity press will be designed to accommodate the anvils and cell assembly that are used in the many off-line Walker type devices throughout the country. These cells have 8 tungsten carbide cubes with edge lengths of 25 mm. Our choice of tooling will be a DIA type design (as in the SPring8 system) which allows diffraction through anvil gaps so that tungsten carbide can be used. The cell will allow 2-D diffraction if transparent anvils are used.

This device will allow the community of synchrotron users to grow to include the many scientists that do these experiments in their own lab. Pressure calibration, phase boundary reversals, kinetic studies will all be enabled for this community.

The large capacity device primarily provides a larger sample volume for a given pressure and temperature than does the current systems such as the T-cup. This larger volume is critical for both ultrasonic experiments and rheology experiments.

Acoustic velocity/emission: Seismology routinely uses acoustic waves to image Earth's interior and to locate earthquakes. In like manner, high pressure laboratory studies can use elastic waves to characterize the sound speed of minerals at high pressure and temperature and to locate sources of acoustic signals in the sample. The former guides us in interpreting seismic velocities; the latter may lead us to an improved understanding of deep earthquakes. Our large-volume beamline at the NSLS was the pioneer to develop tools and results in measuring acoustic velocity at high pressure and temperature. The tools and techniques developed here have been copied around the world, by GSECARS at the APS, and at SPring8 by Irifune. The equipment that we use at X17B2 was purchased by NSF grants for another research program and is on loan from that program. We are requesting funds to purchase a complete ultrasonic velocity system. This will enable us to schedule ultrasonic users at their convenience.

Table: summary of support during last 5 years

Operations	
COMPRES	$330,000/yr
Stony Brook University	$300,000/yr
Facility: Hutch	
NSLS	$1,440,000
CHiPR	$150,000
Stony Brook University	$50,000
Facility: Side Station	
Air Force	$182,000
Stony Brook University	$50,000

Budget Multi-anvil at NSLS

Operations	**2007**	**2008**	**2009**	**2010**	**2011**
Salaries*	$117,000	$120,510	$124,125	$127,849	$131,685
Fringe (36.5%+)	$42,705	$46,396	$50,271	$54,336	$58,600
Salaries + Fringe Total	$159,705	$166,906	$174,396	$182,185	$190,284
Additional Machine shop services*	$82,927	$85,610	$88,373	$91,219	$94,151
Equipment	$15,000				
Supplies/Anvils	$40,000	$40,000	$40,000	$40,000	$40,000
Office space NSLS	$6,500	$6,500	$6,500	$6,500	$6,500
Overhead 33.25% MTDC	$96,136	$99,822	$103,642	$107,601	$111,705
Totals	$400,268	$400,038	$415,347	$431,215	$447,661

* Salaries and machine shop services to grow by 5% per year

Equipment	2007	2008	2009	2010	2011
High Energy/ Resolution Studies	$90,000				
Acoustic emission		$100,000			
Large capacity press w/out DDIA30			$280,000	$302,000	
Ultrasonics					$90,000

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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
Funding requested: $150K for 2007-2012]

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 30, 2006

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 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).

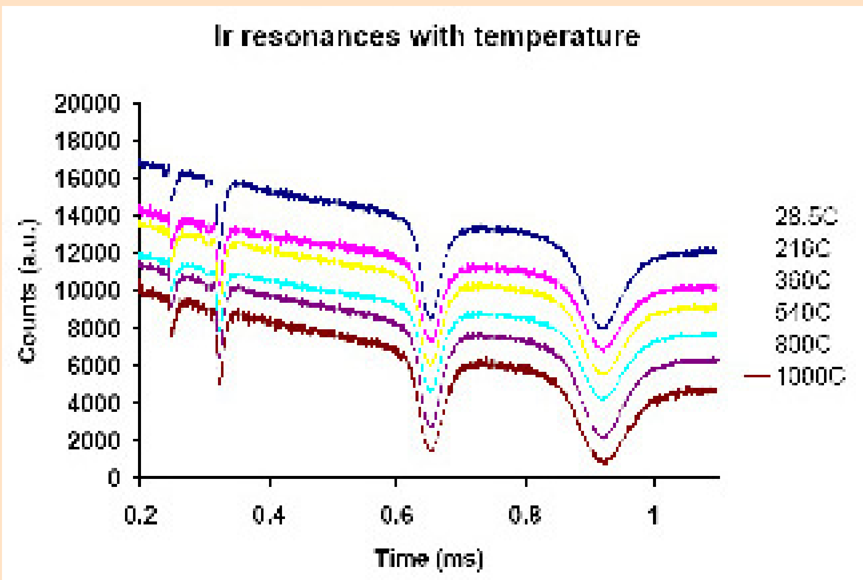

Fig. 1 Plot of neutron resonances of iridium (Ir) between 20°C and 100°C.

Proposal for Neutron Program in COMPRES II

In COMPRES II, the neutron program will continue to play a proactive role in the development of high-pressure neutron research in the United States, but it will be led by individuals who will lead specific projects rather than by N. Ross at Virginia Tech. It is proposed that a COMPRES "task force" for high-pressure neutron research be formed made up members of the COMPRES community, experienced neutron researchers and representatives from neutron sources. The task force will: (i) identify community needs; (ii) encourage exchange of information between high-pressure neutron facilities within the United States (LANSCE, IPNS, HIFR, SNS) and from around the world (especially as J-PARC and the 2nd target station of ISIS come on line); (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. In the transition from COMPRES I to COMPRES II, N. Ross will be responsible for forming the task force and possible members are listed in Table 2. It is important in the first year of COMPRES II that the task force meet to discuss goals, planning and to select a leader. The task force will also communicate by phone and e-mail and will meet when possible in various formats such as at the AGU meetings and COMPRES events.

Below are some projects that the Task Force may consider beyond the first year of the COMPRES renewal proposal:

Table 2: Suggested Members of Neutron Task Force

Bryan Chakoumakos	Hiroyuki Kagi	Darren Locke	Rudy Wenk
David Dobson	Stefan Klotz	John Parise	Yusheng Zhao
Rus Hemley	Martin Kunz	Nancy Ross	
Steve Jacobsen	Kurt Leinenweber	Chris Tulk	

- Purchase and install furnaces in the PE cells of the SNAP project so that high P-T experiments can be carried out. The appropriate furnaces have already been developed at ISIS and it would be straightforward to transfer the technology from ISIS to the SNS. Part of the mission of SNAP is to provide high-pressure capabilities for all beam lines at the SNS, so these cells could be moved to other beam lines for high P-T experiments. Users from the COMPRES community will apply for beam time through a proposal review system. While there is no guarantee of beam time on SNAP, members of the COMPRES community have been very successful in gaining beam time at other dedicated high-pressure beam lines such as PEARL at ISIS.
- Development of an epithermal neutron resonance detector for high P-T measurements at neutron facilities in the U.S. (IPNS (if active), LANSCE, SNS). The neutron task force needs to identify a lead PI for this project and it should be coordinated with existing high P-T calibration efforts in COMPRES.
- The neutron task force should investigate whether it is possible to bring high-pressure capabilities to HIFR on a permanent basis (with ILL as a model). HIFR is being used as testbed for some high-pressure experiments by the SNAP team. If results are encouraging, funds for a workshop may be solicited from COMPRES for development of a larger proposal.
- The SNAP team has identified software development as a major need so that they can immediately start to analyze high-pressure data for ongoing development of instrument and press-specific attenuation corrections, data reduction routines and structure modeling to aid in structure solution of high-pressure phases. Software development is also an issue addressed by other COMPRES initiatives (e.g. CEAD). A breakout session at the 2006 Annual Meeting in Snowbird, Utah was held by CEAD at which input from the neutron as well as the synchrotron community was combined.

The projects above illustrate only a few of areas in which COMPRES can participate in the development of high-pressure neutron science in the United States. There are also opportunities at other sources and beamlines for high-pressure neutron research both in the United States and abroad. There are exciting developments, for example, led by one of our international members (D. Dobson, UCL) using neutrons to study rheological properties of materials at high P and T. In summary, dedicated personnel capable of pushing beyond what has been possible at existing neutron sources will be required for high pressure neutron research to flourish in the United States as it has throughout other parts of the world. COMPRES can play an integral part in achieving these goals by identifying and supporting teams who come forth with proposals that address the needs of the community.

Budget:

$30K is the level of funding required for Year 1 of COMPRES II to cover the costs of a working group meeting of the neutron task force/workshop and travel/subsistence expenses for U. S.-based COMPRES researchers to carry out experiments at high-pressure neutron facilities . It is proposed that these funds be held centrally. In the COMPRES I era, this activity was categorized under Community Facilities; in COMPRES II, it will be included as an Infrastructure Development project.

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2004

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Sitepu, H., J. Wright, T. Hansen, D. Chateigner, H.-G. Brokmeier, C. Ritter and T. Ohba (2005). Combined synchrotron and neutron structural refinement of R-phase in Ti50.75.Ni47.75.Fe1.50 shape memory alloy. Mat. Science Forum **497**: 255-260.Neutron Studies

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Bréger, J., N. Dupré, P. J. Chupas, P. L. Lee, T. Proffen, B. , J. B. a. P. Parise and C. P. Grey (2005). Short- and Long-Range Order in the Positive Electrode Material, Li(NiMn)0.5O2: A Joint X-ray and Neutron Diffraction, Pair Distribution Function Analysis and NMR Study. . J. Am. Chem. Soc. **127** 7529-7537 Neutron Studies

Presentations:

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Sitepu, H. *Combined use of neutron and synchrotron for structure refinement and texture of two stage cubic□trigonal□monoclinic martensitic phase transformation in Ti-Ni-Fe alloy*. Proceedings of the International Conference on Neutron Scattering (ICNS 2005), Sydney, Australia, 27 November to 2 December 2005.

Sitepu, H., Ross, N., Hansen, T., Brokmeier, H.-G. and Chateigner, D. (2005). *Quantitative texture analysis of isostatically-pressed molybdite powders by means of neutron diffraction*. Geophysical Research Abstracts, **7**, 08430.

Sitepu, H. and Wright, J.P. (2005). *Use of neutron and synchrotron diffraction data for structural refinement of R-phase shape memory alloy*. Proceedings of the Spring 2005 Conference on EPSRC-ILL Millennium Projects, ILL Grenoble, France, 25-27 May 2005. Abstract.

B.2.Intrastructure 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

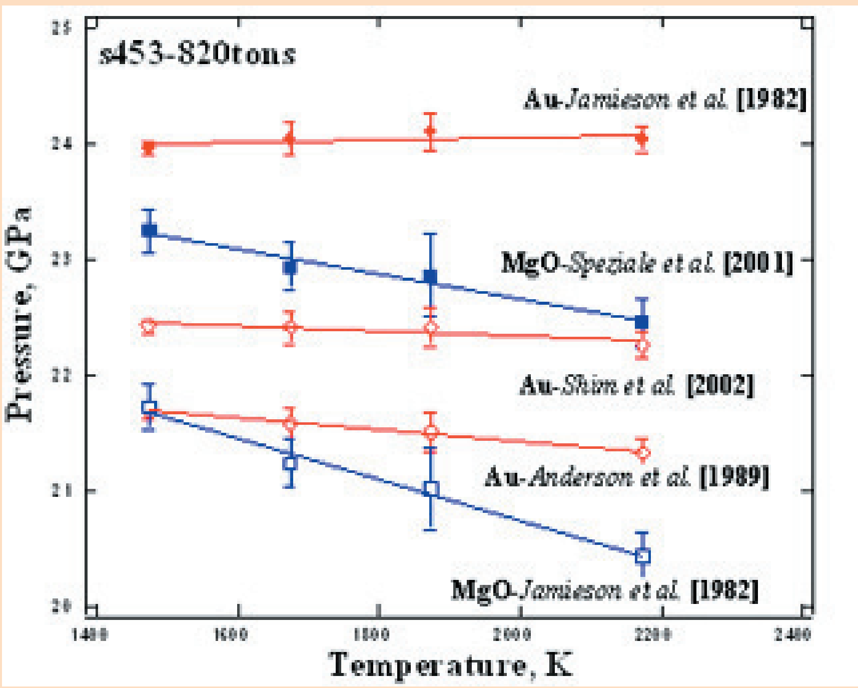

***Fig. 1.** Calculated pressures using MgO and Au pressure scales at high temperatures. Solid circles, open diamonds, and open circles represent pressures calculated from Au scales by Jamieson et al. (1982), Shim et al. (2002), and Anderson et al. (1989), respectively. Solid and open squares represent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

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).

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, Å^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)
Υ_0, K	773	170	230
Υ_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
3*R*, J/gK	0.12664	0.12500	0.12786

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

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References:

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Mao, H.K., Xu, J., Bell, P.M., Calibration of the ruby pressure gauge to 800 kbar under quasihydrostatic conditions. J. Geophys. Res. 91, 4673-4676, 1986.

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Speziale, S., Zha, C., Duffy, T.S., Hemley, R.J., Mao, H.K., Quasi-hydrostatic compression of magnesium oxide to 52 GPa: Implications for the pressure-volume-temperature equation of state. J. Geophys. Res. 106, 515-528, 2001.

Zha, C., Mao, H.K., Hemley, R.J., Elasticity of MgO and a primary pressure scale to 55 GPa. Proc. Natl. Acad. Sci. 97, 13494-13499, 2000.

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
Funding requested for period 2007-2012: $312K

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).

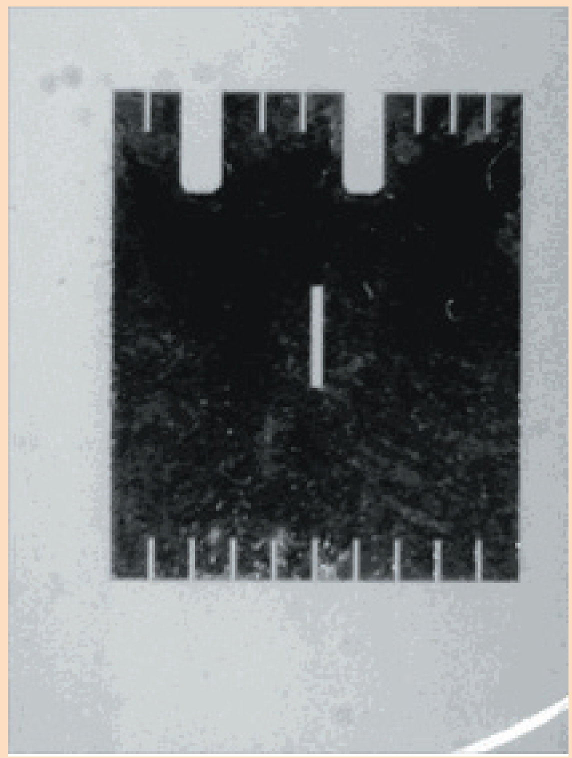

Figure 1. Laser-cut rhenium foil furnace with thermocouple notches, folding contacts, and a central slit for x-ray access.

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 compar-ison. 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).

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

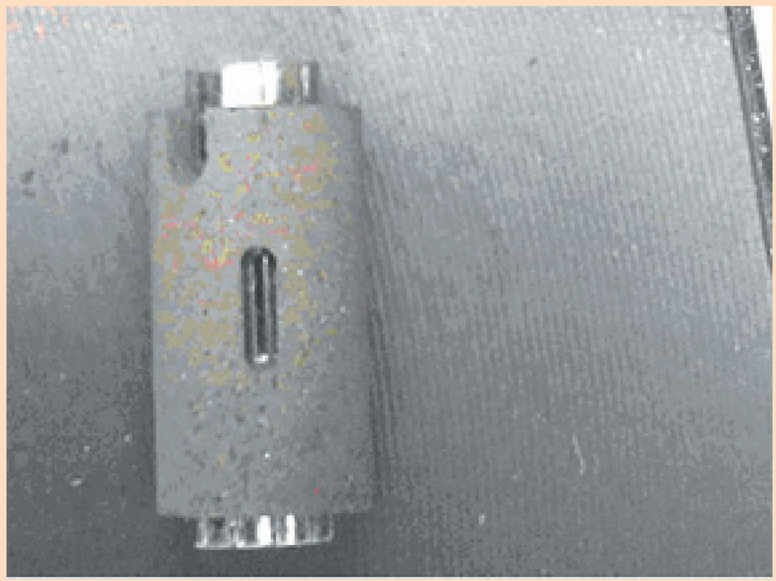

Figure 2. $LaCrO_3$ sleeve with thermocouple slots and x-ray window cut on the COMPRES automated lathe. The slit in the rhenium furnace from Figure 1 is also visible.

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 H_2O to the sample and assembly, which may cause water weakening in deformation experiments, and simultaneously limits

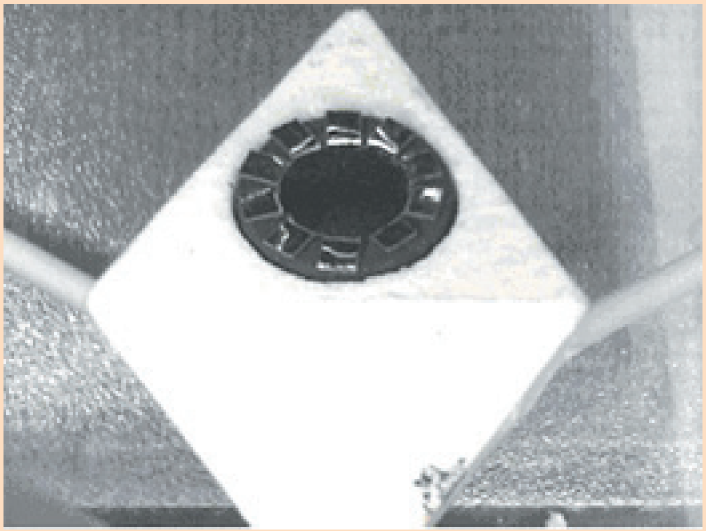

Figure 3. 10/5 in-situ assembly ready to load. The pencil smudge (lower right edge of octahedron) marks the x-ray path.

the temperatures that graphite furnaces can reach. Mullite has good thermal insulation capabilities and is reasonably x-ray translucent, but does not introduce H_2O. This has allowed more reliable dry or controlled H_2O 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 H_2O (11), and the phase boundary determination of the clinopyroxene to ilmenite transition in $ZnSiO_3$ (12).

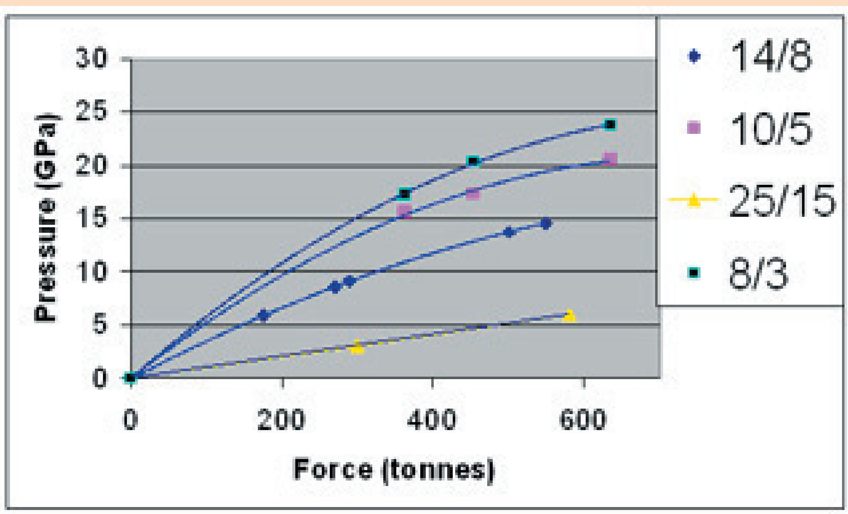

Figure 4: Pressure calibrations of the four conventional assemblies at 1200 °C. Also shown are the preliminary results for the 25 mm octahedron (lowest curve).

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.

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

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 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 mm^3 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.

Publications

2005

Diedrich, T., T. Sharp and K. Leinenweber (2005). Experimental measurements of the effect of water on the kinetics of the olivine to ringwoodite transition in San Carlos olivine. . EOS Transactions American Geophysical Union, Fall 2005. .MAC ASU

Leinenweber, K., J. Tyburczy, T. Sharp, E. Soignard, T. Diedrich, W. Petuskey and J. Mosenfelder (2005). Performance of the COMPRES multi-anvil high-pressure assemblies. EOS Transactions American Geophysical Union.MAC ASU

Mosenfelder, J., K. Leinenweber and Y. Wang (2005). A new multi-anvil assembly for beamline experiments up to 15 GPa: application to in situ measurement of the pyroxene-ilmenite transition in ZnSiO3. . Fall Meet. Suppl., **86(52)**.MAC ASU

2006

Campbell, A. J., L. Danielson, K. Righter, Y. Wang and G. Davidson (2006). Oxygen fugacity at high pressure: equations of state of metal-oxide pairs. Lunar and Planetary Science XXXVII.MAC ASU

Hernlund, J., K. Leinenweber, D. Locke and J. A. Tyburczy (2006). A numerical model for steady-state temperature distributions in solid-medium high-pressure cell assemblies. American Mineralogist **91**: 295-305. MAC ASU

Leinenweber, K., J. Mosenfelder, T. Diedrich, E. Soignard, T. Sharp, J. Tyburczy and Y. Wang (2006). High-pressure cells for in-situ multi-anvil experiments. High Pressure Research.MAC ASU

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/LaCrO3 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

Budget Justification

The primary budget item is the machinist. The machinist performs all of the intensive and specialized ceramics cutting and machining to produce parts for high-pressure cells. In a project that is designed to serve 15 to 20 different laboratories, a large amount of machining is required. The machinist, who specializes in the machining of ceramics, will dedicate full time to this project.

The budget will be supplemented by the sales of standard assemblies at their optimal cost plus a 25% service charge. Machining time and shop usage and equipment are not included in the price of assemblies. The machinist and shop are supplied by COMPRES and ASU, respectively One month of salary per year for the lead P.I., Kurt Leinenweber is included. The tasks of overseeing the purchasing, quality control, and testing of cells, as well as interpreting the feedback from other laboratories, are performed by K. Leinenweber.

The materials request ($4 K per year) is far smaller than the full cost of materials, but the rest will be provided by the community users on a demand basis, and the supply of standard materials will be similar to the demand. The emphasis will be for experimenting with new ceramics and new cell assemblies. This budget will be used to produce experimental parts whose effectiveness is not yet known, and then to supply them, free of charge, to the community for testing and calibration. The travel budget for trips to beamlines ($2K per year) for the *in-situ* testing of the assemblies and for travel to meetings (AGU and COMPRES annual meeting) for dissemination of the results.

BUDGET Summary

	Yr #1	**Yr #2**	**Yr #3**
Machinist's salary	40000	41200	42436
Leinenweber salary (1 month)	5489	5654	5824
Fringe Benefits	14172	14597	15035
Equipment (mini-mill) 9000			
Materials and supplies	4000	4000	4000
Travel	2000	2000	2000
Sub-Total	74662	67451	69295
Indirect costs (49.5%)	32502	33388	34301
TOTAL	107164	100840	103596

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December 14, 2005

Bob

TO: **Prof. Robert Liebermann, President, COMPRES**

FROM: **Michael J. Drake, Head and Director** Mike

SUBJECT: **Multianvil assemblies**

I wanted to take this opportunity to express how pleased we are with the multianvil assemblies we have recently purchased from COMPRES. The assemblies are well designed and well made. Kurt Leinenweber and the other people in charge of this project have done a fantastic job and deserve praise for their efforts.

For over 25 years there has been much talk of interlaboratory standardization of multianvil assemblies and also of multianvil experiments becoming as routine and easy to perform as piston-cylinder experiments. However, with the exception of the development of the Walker module and a few scattered individual efforts, almost no advancement has been made towards these two goals by most of the experimental community. Smaller institutions that cannot afford dedicated machine shops and technicians have been left to rely on using expensive postdoctoral and graduate student time to machine assemblies and assemble runs. The result has been a huge variation in the quality of the multianvil experiments performed in different laboratories, and also by different individuals within laboratories, all accompanied with great waste of financial resources.

The COMPRES program to standardize multianvil assemblies and provide them to the community represents a significant and greatly overdue improvement over this state of affairs. We wholeheartedly support this effort and hope that it will continue and grow in the future.

c. Dr. Kurt Leinenweber
Dr. Kenneth Domanik

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).

Publicatins

2005

Bass, J. D., S. V. Sinogeikin, D. Lakshtanov, V. Prakapenka and G. Shen (2005). Brillouin Scattering and Synchrotron X-Ray Measurements at GSECARS, Advanced Photon Source: Simultaneous Measurements of Sound Velocities and Density. Fall AGU meeting, San Francisco CA.Brillouin

Lakshtanov, D., S. V. Sinogeikin, C. Sanches-Valle, V. Prakapenka, G. Shen, E. Gregoryanz and J. D. Bass (2005). Aggregate Elastic Moduli and Equation of State of B2 Phase of NaCl to 73 GPa by Simultaneous Synchrotron X-ray Diffraction and Brillouin Scattering Measurements. Fall AGU meeting, San Francisco CA.Brillouin

Matas, J., J. D. Bass, Y. Ricard and E. Mattern (2005). Lower mantle structure and composition: insights from generalized inversions of radial seismic profiles. Fall AGU meeting, San Francisco CA.Brillouin

Sinogeiken, S., D. Lakshtanov, C. Sanches-Valle, V. Prakapenka, G. Shen, E. Gregoryanz and J. Bass (2005). Elastic moduli and equation of state of NaCl to 30 GPa by simultaneous x-ray density and Brillouin sound velocity measurements.Brillouin

2006

Sinogeikin, S., J. D. Bass, v. Prakapenka, D. L. Lakshtanov, G. Shen, C. Sanches-Valle and M. Rivers (2006). A Brillouin spectrometer interfaced with synchrotron X-radiation for simultaneous x-ray density and acoustic velocity measurements. Rev. Sci. Instr.Brillouin

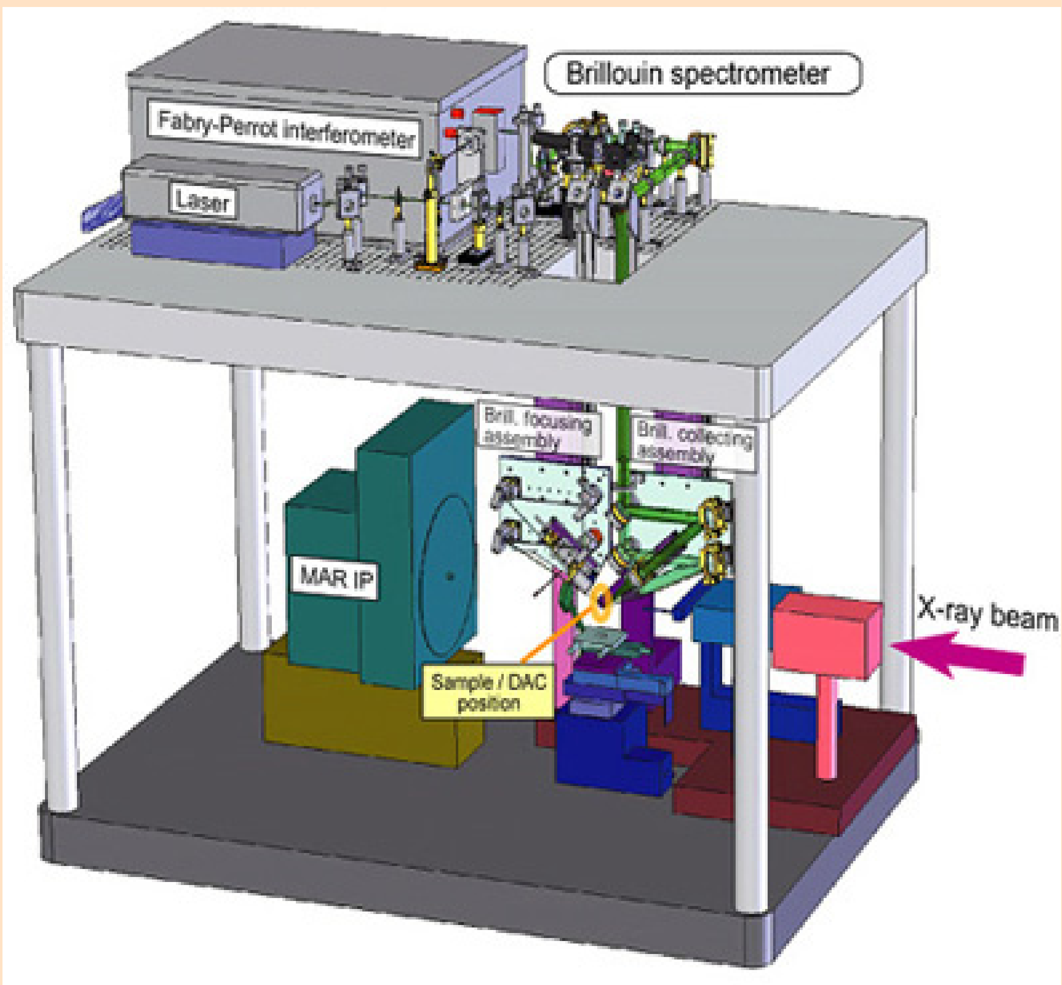


Fig. 1. Schematic diagram of the Brillouin spectrometer at sector 12-BM-D of GSECARS, Advanced Photon Source. Most of the Brillouin optics are on the upper level, whereas the sample is in placed in the x-ray beam for all experiments on the lower level.

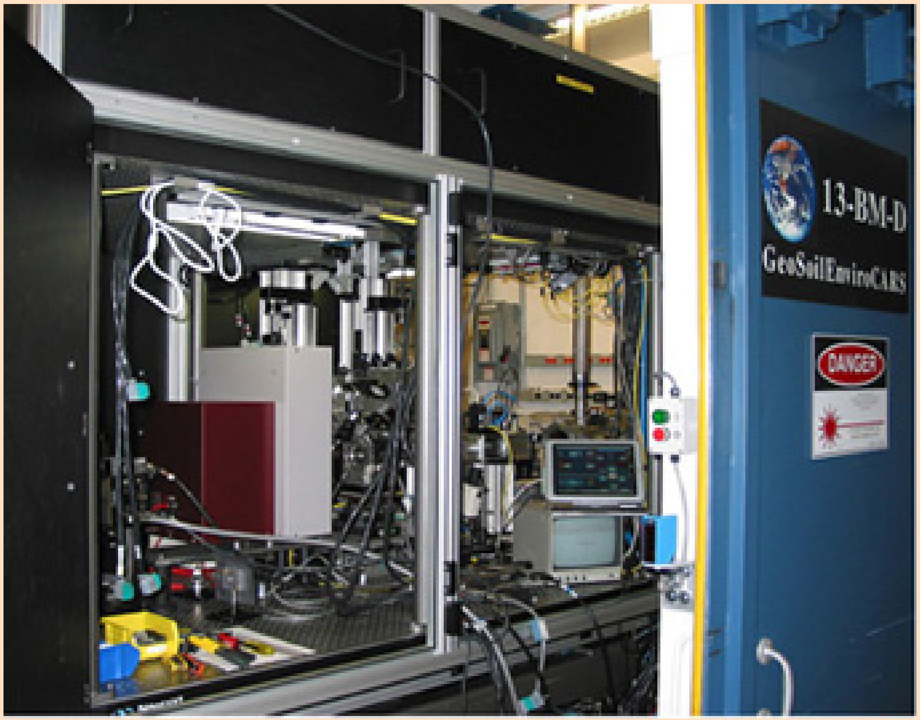
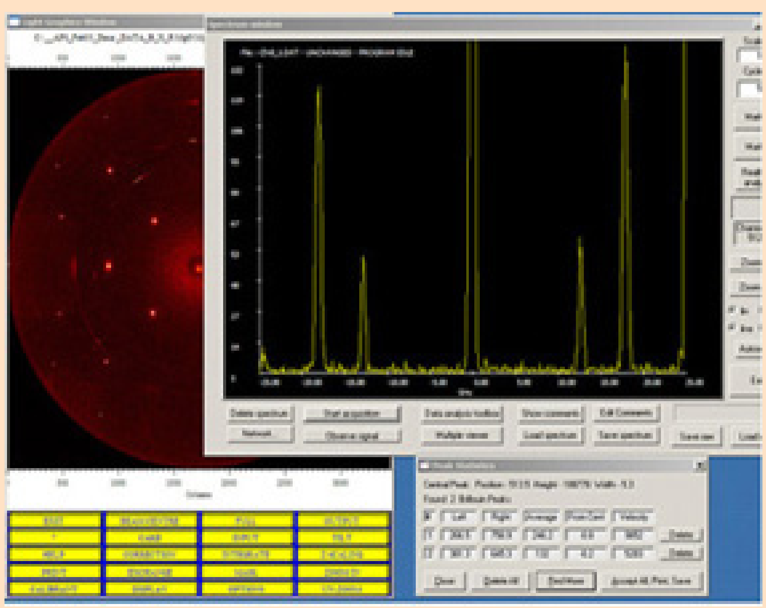


Figure 2: Side view of the Brillouin system at the APS. The upper optics are in the black enclosure at the top of the photo, to comply with APS laser safety regulations.

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



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

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

Current funding for period 2004-2007: $185K
Funding requested for period 2007-2010: $225K

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.

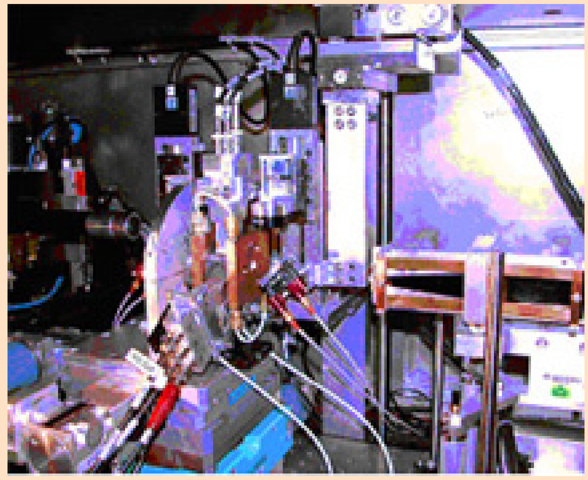


Figure 1b: DAC environment for NRS experiments with laser heating after the addition of clean-up slits. The distance between x-ray focusing mirrors (right) and DAC (middle) was increased, and a clean-up slit assembly was inserted. On the left, a microscope for DAC alignment is visible.

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 follwing websites: http://www.nrs2005.aps.anl.gov andhttp://www.aps.anl.gov/News/Conferences/2005/Mossbauer_Data_Workshop.

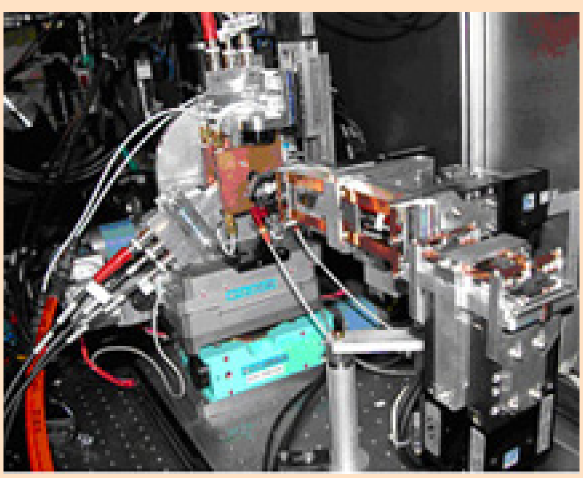


Figure 1a: DAC environment for NRS experiments with laser heating before the addition of clean-up slits.

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.

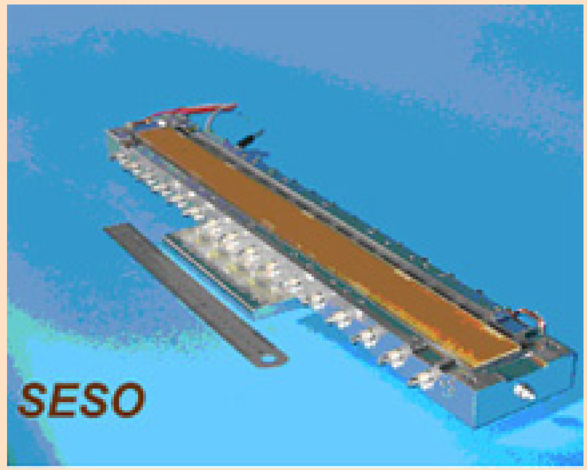


Figure 2: New focusing mirror for high-pressure experimnts at 3-ID. The increase in length from now 20 cm to then 60 cm will provide a significantly higher x-ray intensity for NRS experiments. (Photograph courtesy of R. Signorato, ACCEL, Germany)

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 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. 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 it's own experts to help write proposals, consult on technical aspects of experiment design, and to help run experiments.

Publications

2004

Lin, J. F., Y. Fei, W. Sturhahn, J. Zhao, H. K. Mao and R. J. Hemley (2004). Magnetic transition and sound velocities of Fe3S at high pressure:implications for Earth and planetary cores. EPSL **226**: 33-40.Nuclear Resonant

Lin, J. F., W. Sturhahn, J. Zhao, G. Shen, H. K. Mao and R. J. Hemley (2004). Absolute temperature measurement in a laser-heated diamond anvil cell. GRL **31(L14611)**. Nuclear Resonant

Mao, W. L., W. Sturhahn, D. L. Heinz, H. K. Mao, J. F. Shu and R. J. Hemley (2004). Nuclear resonant x-ray scattering of iron hydride at high pressure. GRL **31(L15618)**.Nuclear Resonant

Papandrew, A. B., A. F. Yue, B. Fultz, I. Halevy, W. Sturhahn, T. S. Toellner, E. E. Alp and H. K. Mao (2004). Vibrational modes in nanocrystalline iron under high pressure. PRB **69(144301)**.Nuclear Resonant

Shen, G., W. Sturhahn, E. E. Alp, J. Zhao, T. S. Toellner, V. B. Prakapenka , Y. Meng and H. K. Mao (2004). Phonon density of states in iron at high pressures and high temperatures. PCM **31**: 353-359.Nuclear Resonant

Struzhkin, V. V., H. K. Mao, W. L. Mao, R. J. Hemley, W. Sturhahn, E. E. Alp, C. L'Abbe, M. Y. Hu and D. Errandonea (2004). Phonon density of states and elastic properties of Fe-based materials under compression. Hyperfine Int. **153**: 3-15.Nuclear Resonant

Zhao, J., G. Shen, W. Sturhahn and E. E. Alp (2004). Highly efficient gaseous sample loading technique for diamond anvil cells. Rev. Sci. Instrum. **75**: 5149-5151. Nuclear Resonant

Zhao, J., W. Sturhahn, J. F. Lin, G. Shen, E. E. Alp and H. K. Mao (2004). Nuclear resonant scattering at high pressure and high temperature. High Press. Res. **24**: 447-457.Nuclear Resonant

2005

Jackson, J. M., W. Sturhahn, G. Shen, J. Zhao, M. Y. Hu, D. Errandonea, J. D. Bass and Y. Fei (2005). A synchrotron Mössbauer spectroscopy study of (Mg,Fe)SiO3 perovskite up to 120 GPa. Amer. Mineralogist **90**: 199-205.Nuclear Resonant

Lin, J. F., W. Sturhahn, J. Y. Zhao, G. Y. Shen, H. K. Mao spin state of iron in minerals of Earth's lower mantle. Geophys. Res. Lett. **32(L12307)**.Nuclear Resonant

2006

Lin, J. F., A. G. Gavriliuk, V. Struzhkin, S. D. Jacobsen, W. Sturhahn, M. Y. Hu, P. Chow and C. S. Yoo (2006). Pressure-induced electronic spin transition of iron in magnesiowustite-(Mg,Fe)O. Phys. Rev. B **73(113107)**. Nuclear Resonant

Published abstracts that so far have not been followed by peer-reviewed publications include:

Shen et al. (2004) Eos Trans. AGU, Fall Meet. Suppl., "Valence state of iron in mantle phases at high pressures and high temperatures"

Li et al. (2005) GSA Annual Meeting, Salt Lake City, UT, "Probe the electronic spin state of iron in lower mantle perovskite through a combination of x-ray emission spectroscopy and synchrotron Mössbauer spectroscopy to megabar pressures"

Jackson et al. (2005) Eos Trans., AGU, Fall Meet. Suppl., "Novel melting investigations of iron at high-pressure using synchrotron Mössbauer spectroscopy"

Jackson et al., (2006) AGU Joint Assembly, Baltimore, MD, "Sound velocities of upper mantle minerals determined by nuclear resonant inelastic x-ray scattering".

Proposal for New Project for 2007-2010

Proposal Summary

Inelastic x-ray scattering techniques with meV energy resolution are relatively new applications of synchrotron radiation for determining the properties of condensed matter. The infrastructure development outlined here is aimed at creating state-of-the-art inelastic x-ray scattering techniques for characterizing the properties of materials under the high-P-T conditions of planetary interiors. The development and advance of two powerful techniques are of interest: momentum-resolved Inelastic X-ray Scattering (IXS) and Nuclear Resonant Scattering (NRS). The applications include (but are not limited to) determining the phonon density of states, phonon dispersions, sound velocities, Grüneisen parameters, phonon softening near phase transitions, viscosity in liquids, and detection of melting, all for iron-bearing (NRS and IXS) and other (IXS) compounds of geophysical interest. The feasibility of the NRS technique at high-P-T conditions has recently been established at sector 3-ID, and it was shown that IXS can be performed using small samples at high pressures in a diamond anvil cell.

We propose to develop a new experimental capability at sector 30-ID of the Advanced Photon Source, to enhance the experimental capability at sector 3-ID, and to make them accessible to the COMPRES community. We will develop laser heating of samples in a diamond anvil cell (DAC) for IXS and NRS experiments. The motivation is to characterize materials by IXS and/or NRS while they are at simultaneous high P-T conditions that are similar to those in the deep Earth. Using the IXS technique we will also be able to study non-iron bearing materials and liquids to address some of the crucial issues in Earth and planetary science not accessible by other methods. Such a capability would be unique worldwide. Thus far, the new 30-ID beam line with micro-focusing capability is under commissioning. The specific tasks to be performed under this proposal are

- Commissioning of the IXS setup for high-pressure experiments at sector 30-ID of the Advanced Photon Source.
- Development of refined high-pressure equipment and techniques for IXS and NRS.
- Development of user-friendly instrumentation and controls to facilitate easy access of the COMPRES community to this new capability.
- Education and outreach to the Earth-sciences community to encourage the use of IXS and NRS, to develop productive collaborations, and to address common experimental issues confronting users.

For the successful and timely completion of these tasks, we request partial funding for a full-time post-doctoral scientist who will be dedicated to developing the high-P-T infrastructure, serving its users, and building a user base among the COMPRES community. We also request funds for workshops and necessary travel expenses. The completion of the tasks outlined in this proposal will

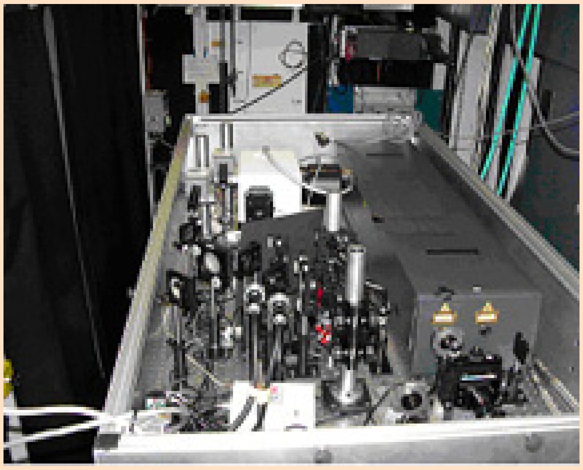


Figure 3: Laser-heating system for NRIXS studies at beam line 3-ID of the Advanced Photon Source.

provide a truly unique capability for the study of vibrational dynamics of deep Earth materials under high pressure and high temperature conditions. No other IXS or NRS facility in the world exists for such experiments. The enhancements of sector 30-ID and 3-ID present an ideal synergy with developments at the beam lines of GSECARS and HPCAT, which focus on diffraction and XAFS-type methods.

Introduction and Scientific Motivation

High-resolution inelastic x-ray scattering techniques provide the Earth and planetary science community with opportunities for new and exciting results on the properties of materials at high pressure and temperature conditions. These opportunities are possible due to the characteristics of third generation synchrotron sources such as the Advanced Photon Source. High-resolution IXS techniques fall into two broad areas. One is Nuclear Resonant Scattering which provides information on electronic, vibrational, and elastic properties, such as the density of states and sound velocities. It is also sensitive to solid-melt transitions. The other class of experiments is momentum-resolved IXS which directly gives the dispersion relation of low-energy collective excitations like phonons. Such measurements provide directional information on vibrational and elastic properties, such as the elastic tensor and sound velocities. Both methods are in many ways ideally or even uniquely suited for addressing a number of important geophysical questions. Several examples are:

Sound velocities and elasticity: A number of laboratories are attempting to measure sound velocities at elevated pressure and temperature, using a variety of techniques. Such measurements allow one to interpret the seismic properties of the mantle and core. This is the primary motivation for IXS measurements of sound velocities. Note the method is suitable for metals and opaque minerals. The pressure range of 10-15 GPa and above is extremely challenging for velocity measurements, and inter-laboratory calibrations are necessary to assess absolute accuracy. Velocity measurements under P-T conditions of the lower mantle are mostly *terra incognita*. With NRS one uses the ^{57}Fe isotope to obtain sound velocities from iron-containing compounds (*Hu et al. 2003*). The viability of this method has been demonstrated with materials under pressures up to 153 GPa and temperatures up to 1700 K (*Mao et al. 2001, Lin et al. 2005*). Momentum resolved IXS has been applied to determine sound velocities at pressures above 100 GPa and to obtain the elastic tensor (*Fiquet et al. 2001, Antonangeli et al. 2004*), and there is no obvious barrier to further application at simultaneous high temperatures.

Liquids: The physical properties of liquids such as viscosity, melting, and compressibility under high-pressure conditions are often poorly understood. However, current models of the Earth suggest that molten materials probably exist from Earth's surface down to the outer core. Therefore, measurements capable of extracting melt properties at pressures and temperatures reaching into the outer core P-T regime would be extremely valuable to substantiate, refine, and/or improve our understanding of Earth's interior dynamics. For example, the momentum resolved IXS technique was used to determine melt viscosities for $T > 3000$ K at ambient pressure (Sinn et al. 2003). The extension of such experiments into high-temperature and high-pressure regime would provide a wealth of new information that is not available today through other experimental methods.

High-spin low-spin transitions: Recent synchrotron experiments indicated a high-spin low-spin crossover (HS-LS) of the Fe in magnesiowüstite at lower-mantle pressures (*Badro et al. 2003, Lin et al. 2005a*). This crossover probably has an important influence on the partitioning of Fe and other elements, sound velocities, viscosity, and density of the lowermost mantle. The temperature dependence of this change of spin state, even though theoretically predicted (*Sturhahn et al. 2005*), has yet to be explored experimentally. NRS techniques are ideal to study such phenomena, e.g., a HS-LS crossover collapses the electric field gradient at the Fe nucleus and thereby the nuclear level splitting (*Lin et al. 2006*). NRS in the laser heated DAC would therefore offer an independent means of studying HS-LS transitions at lower mantle pressures and temperatures.

Melting in Fe: NRS allows one to observe transitions from the solid to the liquid state. In the liquid state, the NRS forward scattering signal disappears. Therefore, the NRS technique is an alternative means of detecting melting that is complementary to more commonly employed x-ray diffraction techniques (*Shen 1998, Boehler 2000, Shen 2001*). The intensity of an x-ray diffraction signal is sensitive to grain growth and preferred orientation of grains, while NRS is not. Moreover, NRS can distinguish between an amorphous high pressure solid (*Hemley 1988*) and a true liquid. NRS should therefore allow one to investigate the melting of Fe and Fe alloys, which are the main constituents of the core. This would help resolve controversies on the temperature of the Earth's core.

These examples are meant simply to illustrate the potential use of high-resolution IXS in the Earth and planetary sciences. We are not requesting support for any particular scientific agenda in this proposal. Our intent is to offer examples of what can be achieved using these techniques, and to provide a sound justification for why COMPRES should consider investing resources in them.

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Development of sector 30-ID and refining high-P equipment

The new sector 30-ID beam line will be dedicated to IXS techniques and will provide a highly intense micro-focused x-ray beam. It is scheduled to become operational and therefore accessible to COMPRES users in 2007. To support high-P-T experiments we will implement automated control and calibration procedures, as well as develop and test operating procedures. The goal is to produce a user-friendly system that may be operated safely with a minimum of support. We also plan to install a laser-heating system similar to the one operational at sector 3-ID of the Advanced Photon Source. The logical conclusion of this effort consists of the full integration of this system with IXS experiments using the DAC. The integrated system will be tested with selected samples. The costs of materials and equipment will be covered by the Inelastic X-ray and Nuclear Resonant Scattering group at the Advanced Photon Source. The group is lead by Sturhahn and operates sectors 3, 9, and 30.

The data collection time for an IXS spectrum of samples loaded into DACs is typically 8-12 hours. Stable heating conditions over hours using samples mounted in DACs have not yet been standardized. Different types of samples (e.g., metals versus minerals), may require different loading techniques using different thermally-insulating pressure-transmitting media. Also, chemical compatibility over the course of several hour runs needs investigation. In short, for laser-heated samples special sample preparation and DAC loading techniques must be developed to ensure long-term stability of a run and high data quality. Equally important, users must have someone to interact with on these issues in order for them to arrive with suitable samples and to efficiently use allocated beam time. Testing of such specially prepared DACs must take place in the x-ray beam. We propose to study this problem with COMPRES user groups to achieve the most sensible approaches. Workshops will be an ideal format for this. New loading techniques using a range of materials will require tests under actual experimental conditions. The development of needed cooling methods for the DACs, as well as studies of their mechanical stability over long time periods will be performed by the requested postdoc.

The successful completion of the tasks outlined in this proposal will provide a new resource to the COMPRES community. It would result in the availability to COMPRES members of momentum-resolved inelastic x-ray scattering techniques at sector 30-ID and nuclear resonant scattering techniques at sector 3-ID using laser-heating equipment. This facility would be used to heat samples in a DAC to simulate the P-T conditions of deep planetary interiors. We want to emphasize that although the sector 3-ID and 30-ID beam lines are not dedicated to high-pressure research, considerable effort has been expended on the installation of infrastructure that is important for diamond cell research on the dynamical properties of materials under extreme conditions. Of particular importance are focusing optics to concentrate the synchrotron beam onto small samples in a DAC and a laser-heating system. This investment can be considered a national treasure, and we hope that the COMPRES community will take advantage of the opportunity it presents.

We propose to hire a scientist at the postdoctoral level to be trained in IXS techniques and their application to high pressure-temperature conditions. This scientist would be the primary contact for COMPRES members who plan to utilize the novel capabilities at sectors 3 and 30. The postdoc would also be responsible for full implementation and commissioning of the high-pressure setup in sector 30. The IXS techniques place special demands on laser-heating technology. In particular, IXS experiments are of very long duration, and a very stable heating system is required. The length of a typical IXS experiment also means that an experienced scientist must be present for long periods of time with inexperienced users. Finally, the postdoc will take the lead in organizing workshops and other outreach to the COMPRES community. The personnel now assigned to sectors 3 and 30 would be unable to handle these additional tasks and to accommodate many new inexperienced users.

Education and Outreach

High-resolution inelastic x-ray and nuclear resonant scattering are relatively new techniques that are not widely available. Therefore, education of and outreach to the Earth-sciences community on these methods is as important as the technical and experimental objectives of this proposal. We request funds for one workshop on Inelastic X-ray Scattering per year of this project. Participants would learn about the capabilities and the theoretical background of IXS methods, visit sectors 3 and 30 at the Advanced Photon Source where IXS and NRS are performed, and obtain some hands-on experience. Workshops will also be an ideal format to collectively address possible solutions to experimental problems. We will work with potential users on an ongoing basis to develop technically sound and scientifically competitive proposals for beam time. If opportune we will prepare articles on the IXS and NRS techniques for the COMPRES newsletter to provide the community with basic information on IXS and NRS and to keep them informed on issues related to this project. We will actively work to build a viable COMPRES user base for this facility.

User Access and Beam Time Proposals

Access to sectors 3-ID and 30-ID beam lines by COMPRES members will be facilitated through the general user proposal system of the Advanced Photon Source. IXS and NRS techniques are novel and few scientists are familiar with proper design and selection criteria for the experiments, particularly under high-P-T conditions. Success in applications for beam time and in actual measurements depends crucially on the information flow between COMPRES user groups, the experts on IXS and NRS, and experts in high-P-T experiments. The scientist to be hired under this proposal will be the linchpin in the development of a COMPRES interest group to utilize the unique opportunities of IXS and NRS under high-P-T conditions.

Budget Justification

Salaries: We request approximately 60% of the salary per year for the postdoc who will be fully committed to this project. This person will take a leadership role in the final design, construction, calibration, and commissioning of the setup for IXS-DAC experiments and for the laser-heating system for NRS-DAC experiments. The postdoc will also be the main organizer of workshops related to this project, as well as shorter user group and informational meetings that may be piggybacked onto conferences such as the AGU and annual COMPRES meetings. The post doc will be the main contact for new users of the high-PT DAC capabilities of sectors 3 and 30, assisting them with proposal preparation for beam time, carrying out experiments, and analysis of data. These responsibilities are a full-time job and cannot be absorbed by the current staff at sectors 3 and 30. This postdoc will be situated at the Advanced Photon Source (APS) and supervised by Sturhahn, with input from the other PIs. The other 40% of his salary will be provided through a cost-sharing match of approximately $20,000 from the APS. The postdoc salary, fringe benefits, and travel are assessed the off-campus indirect cost rate for UIUC.

No salary is requested for the PIs, who are fully supported by their home institutions or for Bass' and Jackson's students who will be supported by other grants.

Travel: We request $3,000/year for the postdoc to attend one major conference (e.g., Fall AGU), two specialized workshops or meetings (e.g., meeting on inelastic scattering techniques), and several trips to UIUC. Funds are also requested for Jackson, Bass, and their students involved with IXS and NRS for travel to the APS to interact with Sturhahn and the postdoc and to assist with tasks being carried out at the sectors 3 and 30.

Year #1 Budget May 1, 2007 to April 30, 2008

Category		**Amount**
Postdoc salary		$50,000
Fringe benefits (37.56%)		$18.780
Total Salary and benefits		$68,780
Postdoc travel	$3,000	
Jackson travel	$1,200	
Bass + student travel	$800	
Total direct cost (off campus)		$72,980
Total direct cost (on campus)		$800
Total indirect cost (off campus, 26%)		$18,975
Total indirect cost (on campus, 53%)		$424
Total cost		$93,179
APS cost share		$20,179
Total request From COMPRES		**$73,000**

Budgets for Years #2 and 3 will follow this model, with $2K additional funds requested per year.

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

CO_2 laser heating system development

A goal of this project is to develop an on-line CO_2 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 CO_2 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 motorizng 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

2004

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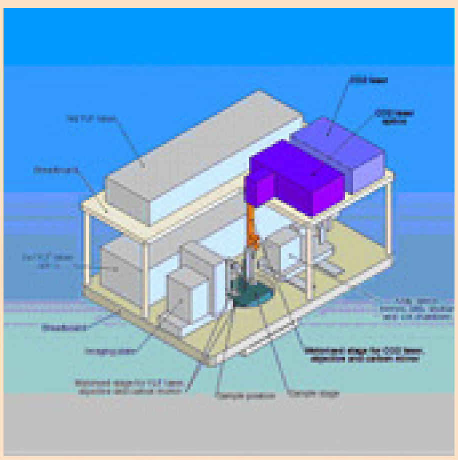

Fig.1. Experimental set-up for CO_2 laser heating in 13-ID-D station of GSECARS.

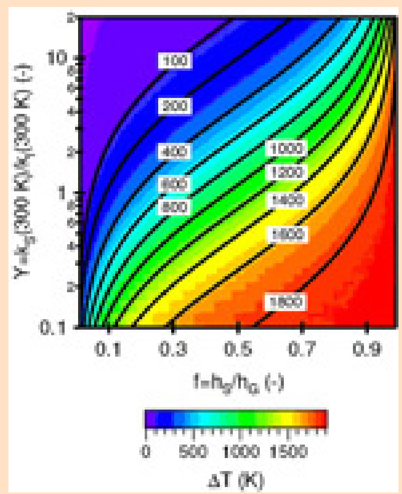

Figure 2. The axial thermal gradient in the laser-heated diamond cell depends on two major factors: the ratio of thermal conductivities of the sample and insulator and the ratio of sample thickness to anvil gap (h_S/h_G). The figure above shows contours of axial temperature decrease across the sample (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

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

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 ìV. 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.

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.

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

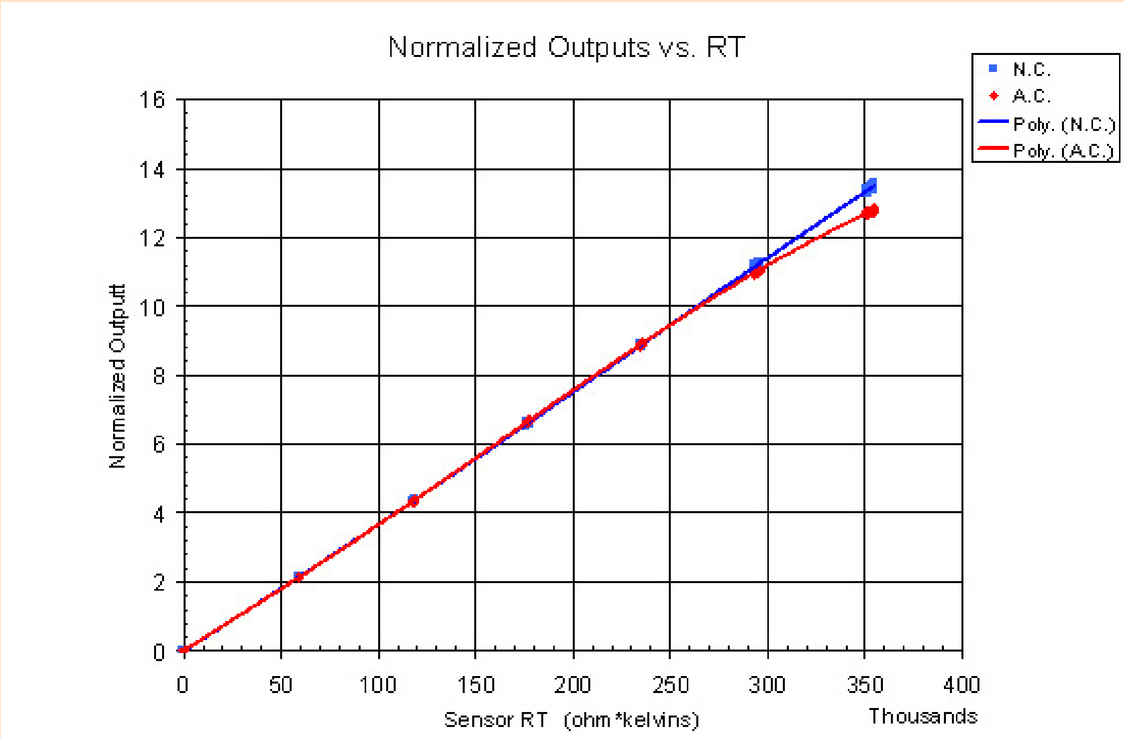

Figure 1. Johnson noise thermometer output vs. the sensor RT product, where R = sensor resistance, T = sensor temperature. In this room temperature study, the sensor resistance is changed in 200 ohm intervals simulating the RT range intended with a nearly constant 100 ohm sensor over a wide temperature range. With the intended sensor, room temperature corresponds to RT = ~50 thousand. RT = ~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.

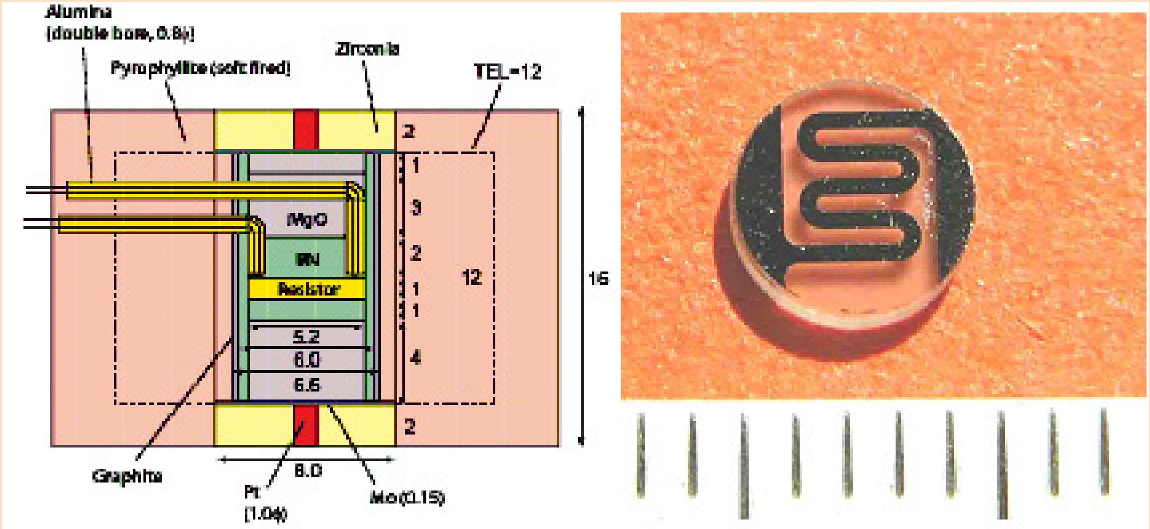

Figure 2. (A) Cell assembly for the first JNT test. All dimensions are in mm. Tests were conducted using the DIA apparatus, with 12 mm anvil truncation. (B) 5 mm diameter resistor for JNT temperature measurements. Ta and Pt layers are vapor deposited on a polished Lucalox substrate to form a mechanically 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.

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 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.

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

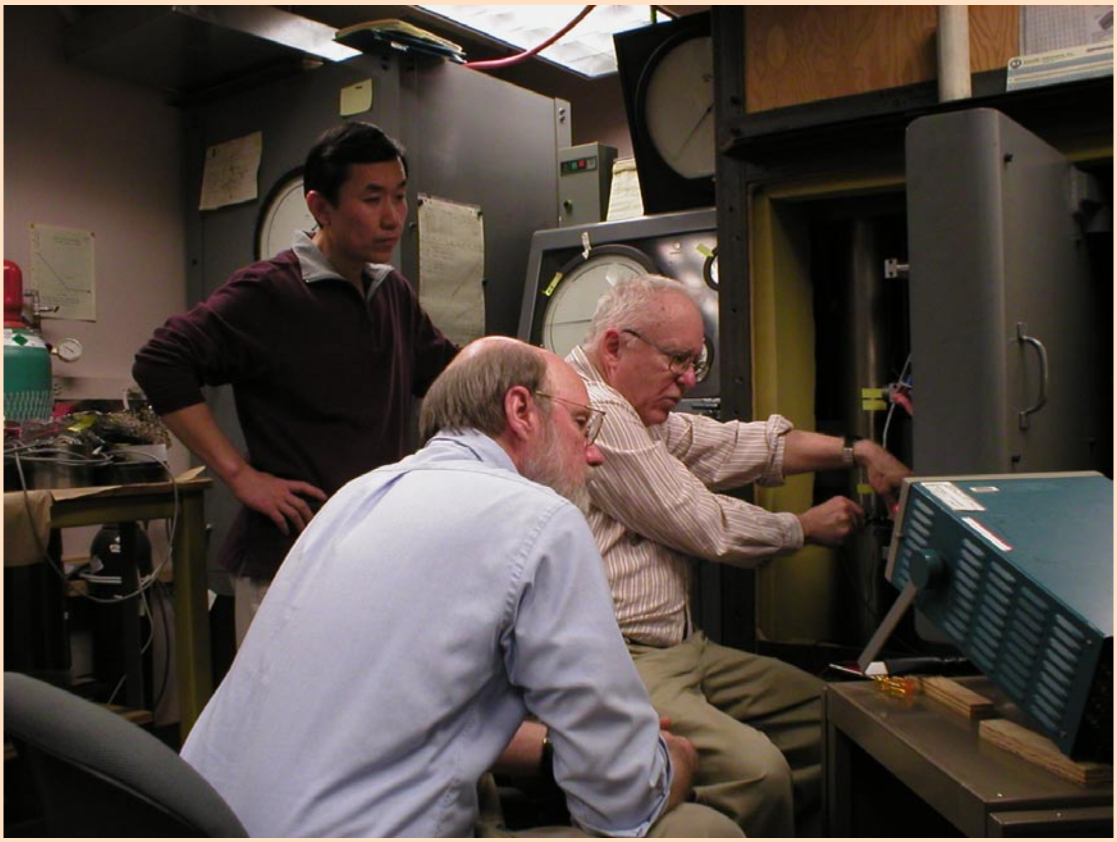

Figure 3. John Hall (right), Ivan Getting (middle) and Yanbin Wang during the high pressure testing of the JNT at U. Colorado, Boulder. Jan., 2006.

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 the period 2005-2007: $170K

The goal of thus project is to develop an automated data analysis environment aimed specifically at the needs of the COMPRES community in a code named CEAD (**C**OMPRES **E**nvironment for **A**utomated **D**ata 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 "hands on" 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

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

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:

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.

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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 15µm long X 5µm 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.

<u>The total funds from COMPRES and NSF are sufficient to support this program for 3 years.</u> 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 ^{o}C.

Workshop

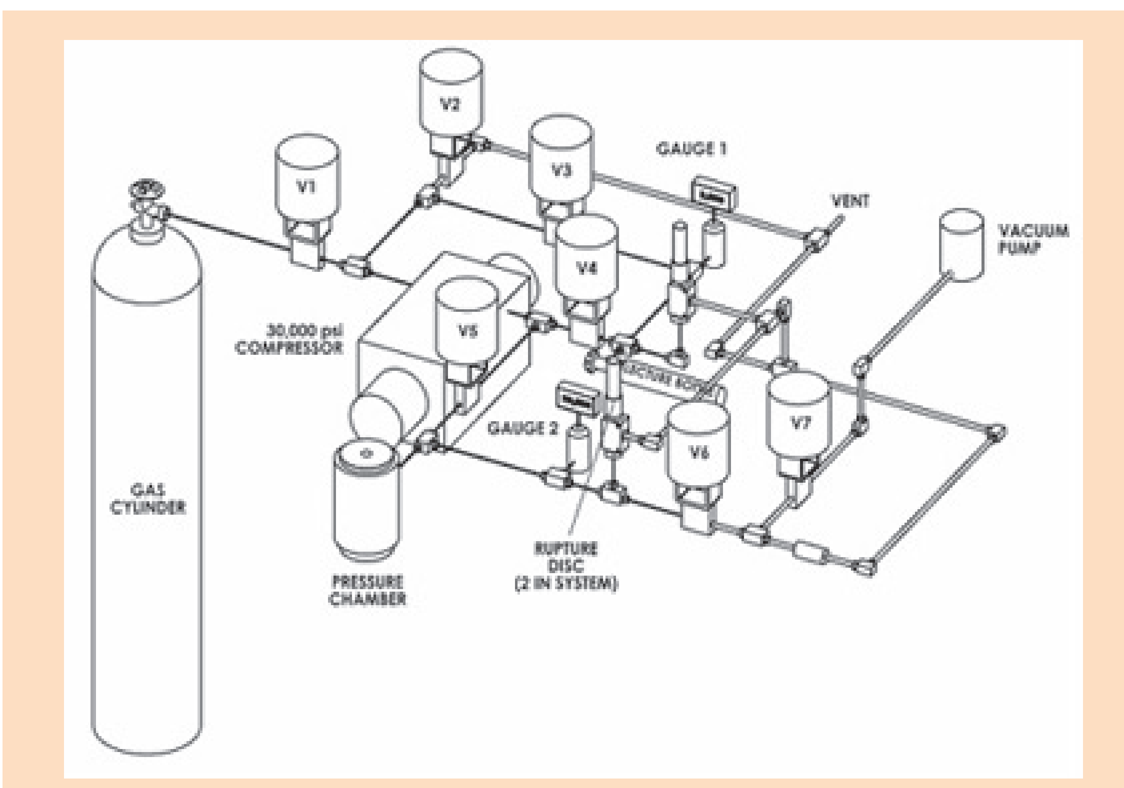
- In order to incorporate input from the community in the design of the system a workshop was held at the APS on April 28, 2006, with 16 specialists from throughout the U. S. attending.

System Design Goals

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

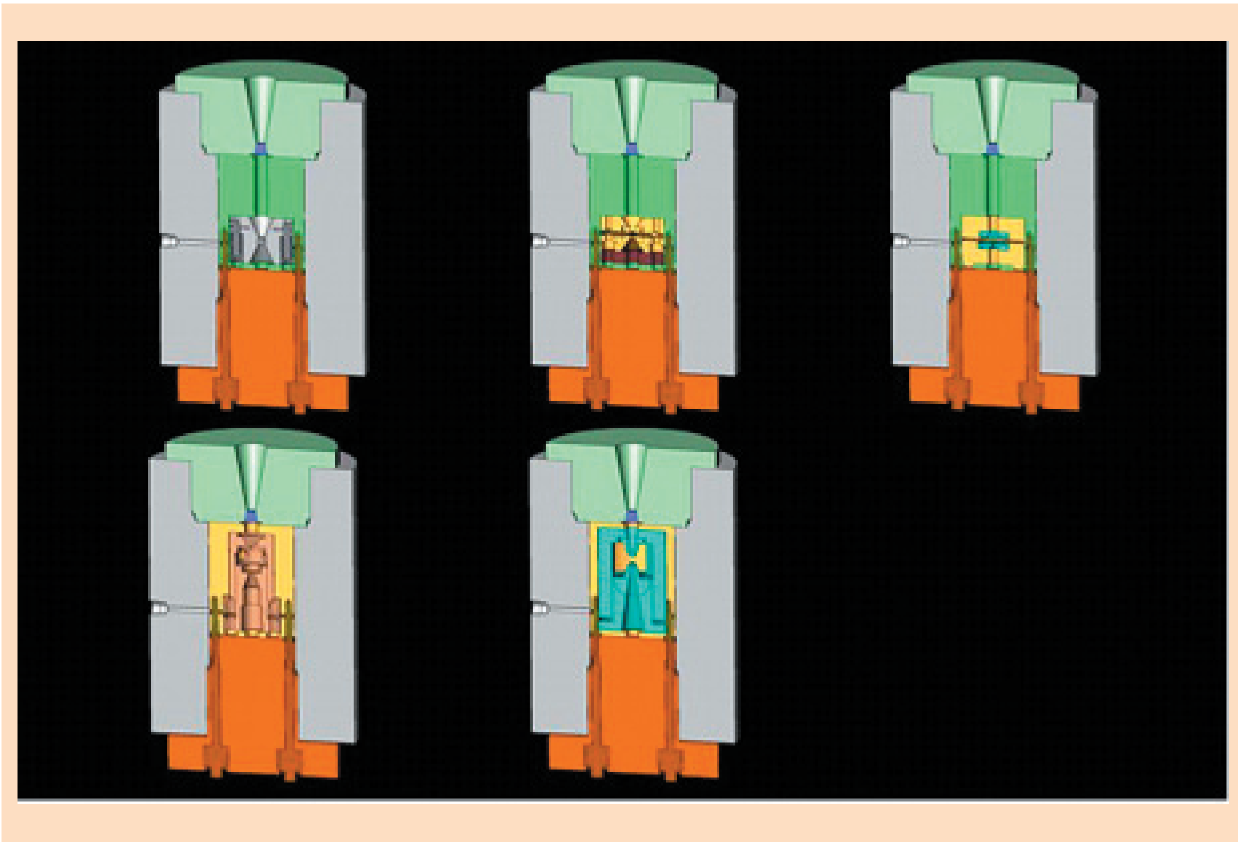
- Able to load many kinds of cells
- Closure mechanism (motor driven screws) will close a clamping device, not the cell itself. Easy to add new cell designs, just a different clamp or different spacers
- Optical access (if possible).
- Vacuum pump to clean system before loading
- No electrical parts 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:

- Gas-operated valves and motor operated drives for remote operation
- PLC for interlock control and computer for routine operations control



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

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. Lesher, 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

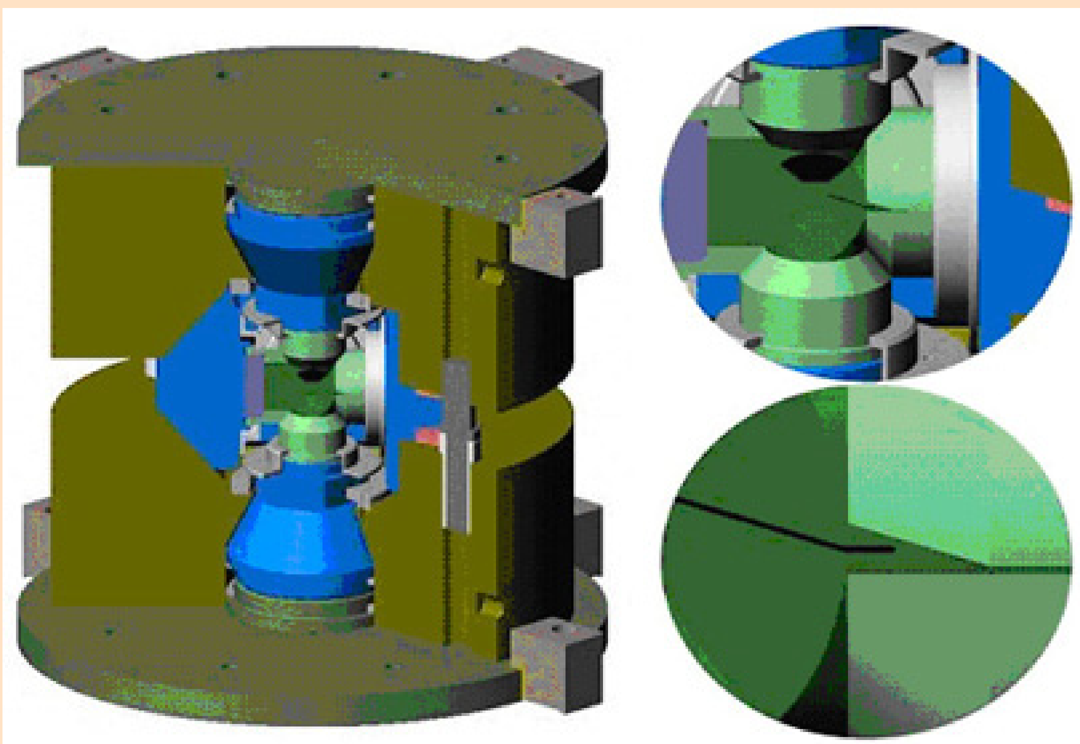


Figure 1. A schematic illustration of the DDIA-30. Horizontal overall dimension 500 mm; vertical height 550 mm. The first-stage DDIA anvils have a maximum of 30 mm truncation, capable of compressing a set of eight 14 mm edge length sintered diamond anvils for 6-8 pressure generation configuration. The 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.

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).

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.

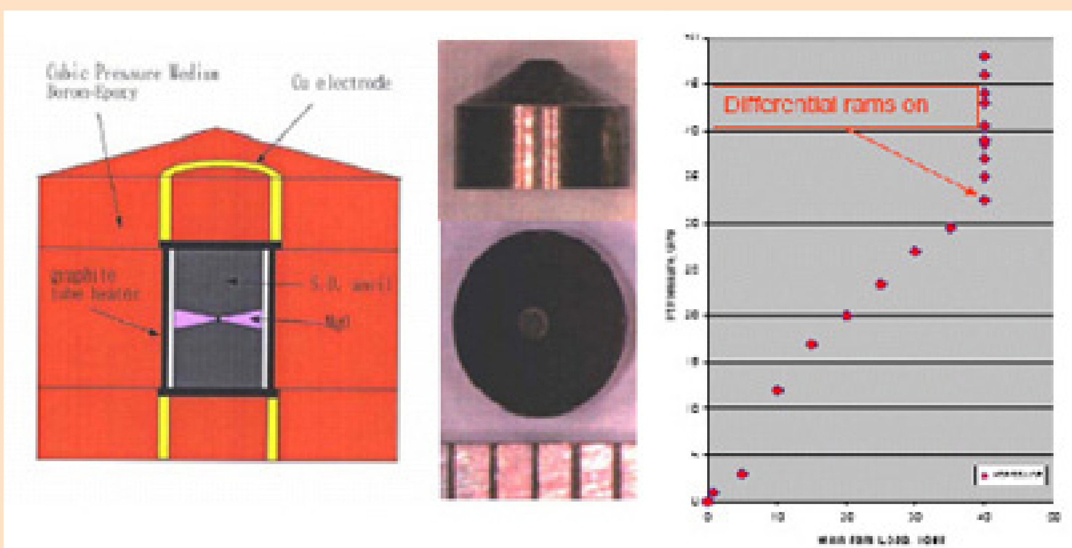
2. A double-stage 6/2 configuration with the second-stage anvils being Drickamer- or diamond-anvil type. With large (10 mm or more in diameter) second-stage anvils, we expect this configuration to generate megabar pressures at high temperature. This configuration has been tested by various groups using 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.

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



A B C

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

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.

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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 Cp measurements. Hellman's microcalorimeters[1] (Figure 1) have been used to successfully measure thin films[2], multilayers[3], and magnetic single crystals[4] in a magnetic field[5] (when applicable) and in a wide temperature range[6]. 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 638μg sample was attached to one of our thick nitride devices (for measuring large heat capacities) with ~100 μg of silver paint. The antiferromagnetic transition is quite well-defined (Figure 2) and agrees with the adiabatic measurement at BYU to within ~1K. This difference is within the precision of our Cernox thermometer. The ~8-10% 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 bulk[7]. 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 Lesher'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 ~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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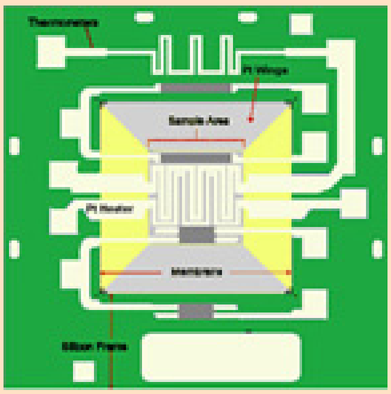
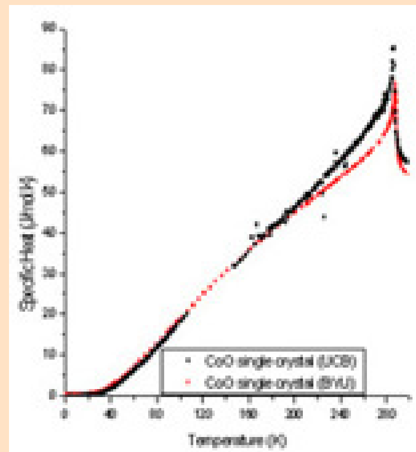
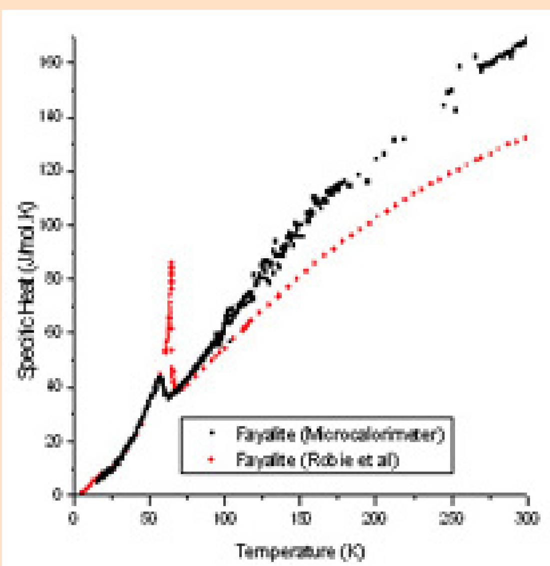


Fig 1. A diagram of our lithographically patterned, silicon-based microcalorimeter.



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



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

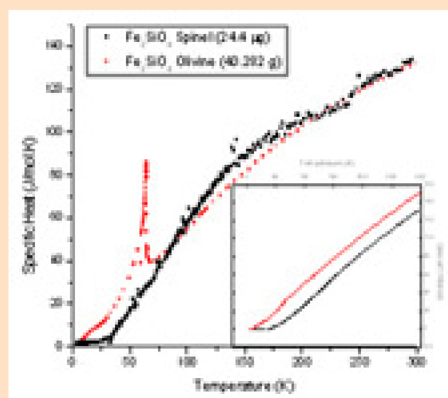


Fig 4. Preliminary results of the specific heat of the spinel polymorph of Fe_2SiO_4 compared to the olivine phase. Entropy information is shown on inset.

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.

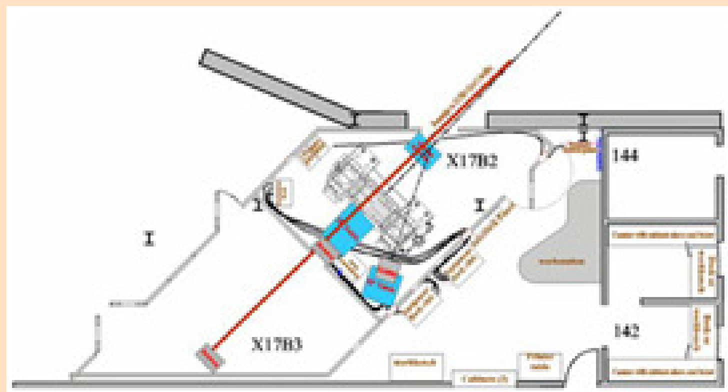


Figure 1. Floor plan of X17B2 and B3 hutches

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.

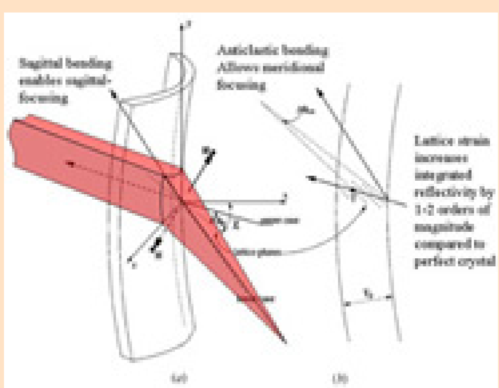


Figure 2. Sagittally bent 2-D focusing monochromator

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 2θ angle to the white beam. A full time monochromatic station running simultaneously with the white beam system is therefore possible.

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.

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.

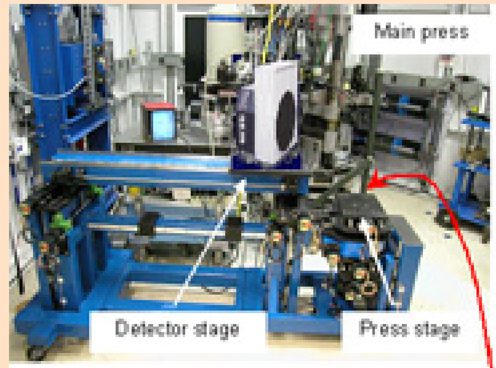


Figure 3. Mar 345 detector and side-station x-y-z tavle in the X17B2 hutch of the NSLS

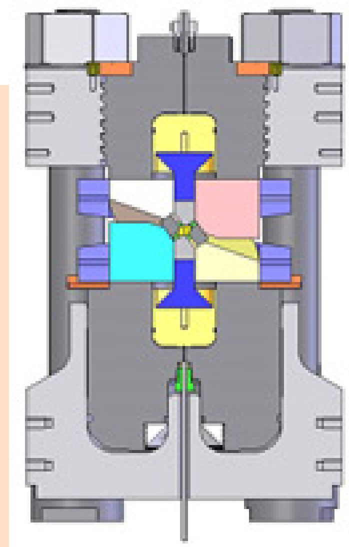


Figure 4. Design of the Deformation-Tcup: a modified Tcup module in a customized Paris-Edinburg press.

B.2.m Commission on High Pressure

[Kurt Leinenweber, Arizona State University]

New Project: Funding requested for period 2007-2010: $150K

Working Group:

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(Note: this list is based on people who clearly expressed interest when this idea was circulated by e-mail. The target number for the Commission should be perhaps 12 to 15 people)

Introduction

The measurement of pressure is a necessary part of all high-pressure experiments. Pressure is measured by means such as separate calibration using fixed points in conventional multi-anvils, *in situ* measurement by ruby fluorescence in the diamond-anvil cell, and by lattice parameter standards in high-pressure diffraction measurements. The effect of temperature on the pressure measurement standard also needs to be known when the experiment involves heating, and in some cases (such as ruby fluorescence) the standard becomes less useful at higher temperatures, and alternative standards need to be found. Good pressure scales are necessary to evaluate the pressures and temperatures of phase boundaries for application to the seismic discontinuities in the Earth, as is shown in efforts such as those by Hirose et al., 2001 and Fei et al., 2004B. It is most desirable to have pressure scales that are derived from physical measurements or theory directly, and are not referenced to any point within the Earth from geophysical observations; otherwise a kind of circular reasoning is suggested, and the use of Mineral Physics as an independent tool for studying the Earth is hampered. Selecting a preferred pressure scale because it gives good agreement with an assumed model of the Earth is an example of this and should be avoided.

Recently there has been a significant effort to improve the accuracy and precision of pressure measurements to address these issues. With the advent of elasticity measurements at high pressure and temperature, in the diamond-anvil cell by Brillouin spectroscopy and Gigahertz interferometry, and in the large-volume apparatus by ultrasonic measurements combined with *in-situ* sample length determination and stress measurement and control, several workers in the field have derived new primary pressure scale (Ruoff et al., 1973) by fitting the elastic moduli and volumes to P(V, T) equations of state. These measurements are ongoing in many laboratories around the world, particularly at room temperature (cf. Bassett et al., 2000; Zha et al., 2000; Mueller et al, 2003; Sinogeiken et al., 2005), and the indications are that the available measurements will increase greatly in the near future. Some studies have even used their own samples as internal scales and worked at high temperature, and have found significant differences from the established pressure scales (Li et al., 2005). Simultaneously, diffraction pressure scales have been intercompared directly or measured relative to the ruby fluorescence scale (Speziale et al., 2001; Akahama et al., 2002; Fei et al., 2004A). Although each group is highly capable of developing pressure scales, a mechanism is needed for reviewing and comparing the various scales, and making recommendations to high-pressure researchers about how the scales are related and what the expected uncertainties are in the scales. This is an issue that vexes practitioners in mineral physics and confuses those who would like to use our data to address geophysical problems, and it needs to be addressed seriously. Because of the expanding base of newer and better data, due in part to breakthroughs in accurate unit cell volume measurements (King and Finger, 1979; Angel et al., 1997) and the availability of recently improved techniques such as multi-anvil ultrasonics and the simultaneous Brilloun/X-ray installation developed by COMPRES, there is now a growing need to make an independent effort to evaluate pressure scales worthwhile.

This proposal creates a new Commission within COMPRES whose task will be to assemble information on pressure scales in just such an independent fashion, and to make recommendations regarding the measurement of pressure in high pressure-temperature experiments. The primary goal will be to gather the pressure scales, evaluate them, and to assign a statistically derived uncertainty to the pressures measured using each one (or to declare that task impossible if the data are insufficient). Communications with the original authors will be initiated when needed to clarify the published results, a pursuit that will be helped by the official status of the Commission within COMPRES.

Approach

As an example, the uncertainty in a primary pressure scale obtained at room temperature from ultrasonic measurements and volume measurements should be derived by propagating the scatter in each dataset through the equation of state that is used. This involves first choosing an independent variable (for example, the wavelength of the ruby fluorescence line in a DAC experiment), which is considered not to have any error. Then, the first and second derivatives of the pressure with respect to every variable in the equation of state have to be evaluated, and the matrix of second derivatives inverted to find the error in pressure as a function of volume. The matrix inverse also reveals all the correlations between different fitting parameters that contribute to the uncertainty. This information is important in evaluating the reliability of a pressure scale, and also in deciding on future experiments targeting the most important parameters that could lead to improvements.

This treatment assumes that the equation of state is correct, and also that there is precisely zero uncertainty in the independent variable. The effect of these assumptions on the measurement of pressure should also be assessed, although this is a more difficult task.

The benefits of having a commission to do this are that it will allow a well-thought-out evaluation of the pressure scales to be done, with diverse methods and opinions brought to bear on each one, and that in the cases where sufficient data is not included in the paper, a commission might have the legitimacy to obtain some of the original data from the authors for analysis. The identification and evaluation of pressure scales would happen through searches and individual or group efforts within the commission. In each case it would be decided whether a scale is independent or is a secondary scale based on an existing one. In the former case, a formal error analysis would be performed, while in the latter case, the uncertainties from the original scale would be propagated into the new one.

An independent check on the scales will be from cross-comparisons of them that will also be ongoing in the experimental community as the scales are developed. These cross-comparisons consist simply of collecting x-ray data for the volumes of several standards in one experiment, calculating the pressure indicated by each standard, and comparing. The values obtained during cross-comparison should fall within the uncertainties of each scale; if they do not, then either the scales or the error estimates need to be re-evaluated.

Ideally, what should exist is a number of independent scales, all of which have been cross-compared as an independent test, and also a number of secondary scales derived from the primary scales. This is a far more realistic goal than trying to come up with a “master” scale and derive all other scales from it – a prescription that would suffer from the additional drawback of having the errors amplified, because the errors from the original scale would be propagated into the derived scales. It will be up to the COMPRES commission to evaluate all the scales in an independent fashion and present them in a final, useable form with their uncertainties.

Operation of the Commission

The commission will communicate by phone and e-mail, will meet in various formats such as at the AGU meetings and COMPRES events, and will organize AGU sessions where the issues of pressure measurement can be discussed by larger groups and combined with talks and posters on new experimental measurements. At times, experts in the creation of scales (such as representatives of NIST and others) will be invited to assist the Commission.

The commission will also coordinate with the Elasticity Grand Challenge, which has a heavy component of pressure calibration experiments. The connection of both of these efforts to COMPRES affords an opportunity to share information. In one sense, it seems that the analysis of pressure scales could be done through the Elasticity Grand Challenge. However, because there are other efforts both in the U.S. and worldwide to develop pressure scales, a commission that is independent of the Elasticity Grand Challenge and consists of a large number of people (including some from the Elasticity Grand Challenge) will be desirable.

The results of this will be published in monograph or other form. In addition, the pressure scales and their uncertainties will be made available as subroutines or modules that can be put into computer programs used to analyze data, including the propagation of the volume error into the pressure measurement.

Targets

COMPRES is assisting in this initiative immediately by providing an opportunity for a breakout session on one of the afternoons at the Annual Meeting in Snowbird, Utah on June 20-23, 2006. This breakout session, to include a broader representation of the community than the list of current collaborators on this proposal, will enable us to formulate a specific plan for a working group/ workshop in the near future to pursue tangible objectives. The discussion will focus on developing a series of targets and planning the initial actions of the group. A preliminary outline of likely targets that can serve as a starting point for this discussion is presented here:

In year 1, the Commission will work mainly on the room-temperature pressure scales, reviewing the origins and the current status of the ruby fluorescence scale, and the physical data (elasticity and volume) for all the common diffraction pressure standards, including MgO, Au, NaCl, and quartz (used in single-crystal studies), and the less common ones such as CsCl and W. A session at the first Fall AGU will be organized on this topic, in which researchers in this field can present their results (topics looking ahead to year 2 and 3, such as high temperature, shock and theory should also be welcome). A monograph on the room temperature pressure scales will be planned for the following summer, which will require a meeting of the Commission, perhaps at Spring AGU.

In year 2, the pressure scales at higher temperatures will be tackled. The diffraction standards will be followed to higher temperature, and the high-temperature fluorescence scales that replace ruby will be examined. The former will require agreement on how to treat the coupling of temperature and pressure in the equations of state. The topic of pressure scales at higher temperatures can be aired at the Fall AGU in the second year of the project. Of course, during this process, new data about the room-temperature scales will appear, and we will continue to worry about the room-temperature scales, because they form the anchor for the high-temperature scales. This also should be published in a monograph form in the summer of Year 2.

In year 3, the detailed consideration of other types of input, shock data and theory, for instance, will be undertaken. Equations of state derived from theory, such as the equation of state for Au developed by Tsuchiya (2003) and in use in Japan, will be considered. These will of course be discussed in years 1 and 2, but the mixing of different types of input into the pressure scales, and extrapolations of the pressure scales outside of the data range, would be best considered later in the work of the Commission, because these problems are more complex and will require a great deal of interpretation.

Other considerations

There were suggestions within the current group to include theoretical and shock people and data, which implies more developed equations of state bringing in several sources of input parameters (and therefore several sources of error and more complex mixing of the errors). The question was also raised as to whether any experiments would be involved, or solicited, although this could tarnish the independence of the Commission from particular experimental results. These possibilities and issues will also be explored by the Commission.

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Budget Request

$30 K per year for dissemination, travel, workshops, meeting events, and possibly experiments.

Year 1	Year 2	Year 3
$30,000	$30,000	$30,000

All budgeted under Participant Support costs, which do not incur indirect costs.

These funds to be held centrally and distributed as needed.

B.2.n High Pressure Synergetic Center at the APS

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

New Project: Funding requested for period 2007-2010:$900K

Project Summary

We propose to establish the High-Pressure Synergetic Center (HPSynC) team to serve the U.S. high-pressure research community of which approximately 50% are Earth scientists. The HPSynC operates on a novel infrastructure concept that connects the large national facility and the high-pressure community. Its main functions are as follows.

1. To integrate high-pressure techniques with specialized synchrotron beamlines to innovate new capabilities.
2. To integrate high-pressure techniques with general synchrotron beamlines at the Advanced Photon Sources (APS) for high-efficiency high-pressure experimentation.
3. To help the high-pressure Earth materials community to increase the total available beam time by >90 days/yr (in addition to the GSECARS beam time).
4. To establish and operate a world-leading high-pressure user laboratory and provide on-site technical support.
5. To introduce new researchers and students to the high-pressure field by organizing training programs and workshops.

The proposed HPSynC team consists of 3.5 scientists (including a part-time project manager), 2 engineers, and an office coordinator. Analogous to the concept of beamline scientists empowering non-specialist researchers to use highly specialized synchrotron facility, HPSynC staff scientists and engineers shall help geoscientists to conduct high-pressure experiments without the preoccupation of technical difficulties, thus enabling the exploration of their scientific agenda to the fullest extent. Unlike beamline scientists whose primary responsibility and resource are limited to individual beamlines, the HPSynC staff access, interface, and optimize high-pressure technology to many beamlines at APS. The HPSynC infrastructure provides a cost-effective leverage to the synchrotron facility and high-pressure community, thus leading the advancement in multidisciplinary high-pressure science and technology.

Introduction

During the past decade, we have witnessed an unprecedented surge in high-pressure research that is improving our fundamental understanding of the Earth's deep interior. The research has greatly benefited from the integration of high-pressure, high-temperature instrumentation and analytical probes at national facilities. For instance, the integration of high-pressure techniques with synchrotron x-ray diffraction and spectroscopy at individual beamlines such as GSECARS, HPCAT and XOR has yielded key information on crystal structure, magnetism, phonon, and electronic dynamics of materials in the lower mantle and core.

The integration has gone a long way. During the first generation approach in 1980's, high-pressure researchers brought the entire experimental setup to an empty synchrotron station each time during very limited beam time allotment. Experiments were restricted to simple ones that could be assembled, aligned, and dissembled within the allocated time. During the second generation since 1990's, dedicated high-pressure beamlines establish permanent setups that allow users to conduct sophisticated high-pressure experiments efficiently. This mode of operation has been very successful; dedicated high-pressure beamlines such as GSECARS and HPCAT have become two of the most productive (in terms of quality and quantity of publications and scientific impact) physical sciences beamlines at APS and around the world.

Dedicated high-pressure beamlines also have noticeable shortcomings. First, with the demand greatly increased dramatically by the success, available beam time at these beamlines is in shortage and oversubscribed by a factor of three. Building additional beamlines to fill the shortage would be very costly. Secondly, many cutting-edge synchrotron techniques that have been advancing rapidly at specialized beamlines are unavailable at dedicated high-pressure beamlines which much consider their primary role in supporting routine, general, high-pressure operations. Users who try to develop or use novel high-pressure techniques at these specialized beamlines on their own are facing the insurmountable barrier of adapting the high P-T conditions to the beamlines analogous to the first generation mode 20 years ago, except it is even harder now due to the sophistication of the advanced beamlines. Meanwhile, the in-laboratory, high-pressure technology has also been advancing at breathtaking rate. Far greater potential in breakthroughs are apparent if we can advance to the third generation integration of high-pressure techniques with all beamlines of the facility.

The Third-Generation Integrated Facility

Ideal integration should be guided by specific scientific goals and should include all applicable techniques, namely synchrotron, neutron, electromagnetic wave, electron, and ion radiation and transport probes together with high-P, high-low-T, and high-magnetic field environments. APS has the largest subset of the capabilities and is a natural place to start the integration. Once the advantage of this mode of operation is widely recognized, this mode may spread throughout other facilities, in a manner analogous to our initial establishment of a dedicated synchrotron beamline twenty years ago and spreading worldwide now.

The need for a coordinated, facility-wide, “extreme environment” infrastructure is not new. In fact, ESRF and SPring-8 already have such setups. But they have been limited to the level of sharing gas-filling equipment, sample preparation laboratory and technician’s helps. While these are important functions, we must progress to the next level of scientific and technical integration. High-pressure scientists who have the scientific agendas and technical know-how in high *P-T* instrumentation must build the infrastructure to fully utilize the extraordinary capabilities and resource of the synchrotron facility.

Such integration can be illustrated by the example of high-pressure nuclear resonant x-ray spectroscopy (NRXS) at APS Sector 3 where Ercan Alp and Wolfgang Sturhahn developed the world-unique NRXS capability. In 1998, Dave Mao saw the potential of using NRXS to obtain key information on shear-wave velocity, heat capacity, Debye temperature, Grüneisen parameter, and Lamb-Mössbauer parameter of Fe at the Earth’s core conditions that are critically needed for understanding the geophysics of the core, and he applied General Users Proposal (GUP) beam time at Sector 3. Consulting with Alp and Sturhahn, Mao designed a new panoramic DAC with Be gasket specifically for NRXS, and borrowed K-B mirror from Peter Eng of GSECARS to focus x-ray beam for megabar NRXS experiment at Sector 3. The experiment succeeded after three years, and resulted in two major publications (*1, 2*). Alp and Sturhahn were very encouraged by the high-pressure Earth science application and continued the collaboration by acquiring dedicated K-B mirrors and a laser-heating system (a total of $200 K), and Mao provided the technical expertise by sending postdoc Jun-Fu Lin to develop the laser heating system. Lin published several seminal papers with this technique (*3-5*) and established NRXS as an important tool in high-pressure geophysics. Sector 3 continued to devote considerable resources (several $M) into this project by adding two undulators, longer bimorph K-B mirrors, and an additional beamline scientist. In 2004, COMPRES funded Sector 3 with a postdoc for reaching out to the COMPRES community.

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2. V. V. Struzhkin *et al.*, *Phys. Rev. Lett.* **87**, 255501 (2001).
3. J. F. Lin *et al.*, *Geophys. Res. Lett.* **30**, 10.1029/2003GL018405 (2003).
4. J.-F. Lin *et al.*, *Geophys. Res. Lett.* **31**, doi:10.1029/2004GL020599 (2004).
5. J.-F. Lin *et al.*, *Science* **308**, 1892 (2005).

This successful example (and a similar example on the development of x-ray emission spectroscopy for studies of high-spin-low-spin transition) also reveals several important lessons.

- Specialized APS beamlines like Sector 3 have the synchrotron expertise and resources and is eager to collaborate, but rely on the high-pressure community for scientific agendas and high-pressure expertise.

- Dedicated high-pressure sector, such as GSECARS, must focus on its primary mission of beamline operation, and cannot play a major role in projects at other sectors.
- In the absence of infrastructure, individual scientists who have excellent ideas but less access to synchrotron beamline and high-pressure equipment than Mao will have an extremely difficult time to carry through such development.
- Neither Mao nor Lin could regard this project as their primary mission. The development is necessarily serendipitous and haphazard in nature. For each successful project, there are at least three stalled or failed projects due to the lack of infrastructure and commitment.
- The development could be much more cost and time efficient, and the success rate could be much higher if there were organized infrastructure and support.

High Pressure Synergetic Center

To connect the missing link, we are organizing the High Pressure Synergetic Center (HPSynC) at APS. The intended community includes all interested high-pressure research scientists in the U.S.; initial contacts indicate the total number may exceed 200. Approximately 50% are Earth scientists; many work at COMPRES member institutions.

HPSynC CoPIs have discussed the plan with APS management and individual beamline scientists at group meetings and APS-wide workshop. They have received enthusiastic supports. The scientific success of high-pressure programs at Sectors 3 (XOR), 13 (GSECARS) and 16 (HPCAT) are very attractive to other beamlines; at least twenty beamlines show strong interests in high-pressure projects. APS management agrees to provide necessary laboratory space and beamline instrumentation to coordination with the high-pressure development.

HPSynC Management Plan

1. Principal Investigators

PIs are responsible for the scientific and technical management of the HPSynC. CoPIs Ho-kwang Mao and Russell J. Hemley will participate in executive issues on monthly basis. CoPI Guoyin Shen, the current HPCAT Project Manager, will share the part-time Project Manager responsibility at HPSynC.

2. Steering Committee

Following GSECARS successful model, we will establish a Steering Committee, consisting of at least five members, including prominent synchrotron and high-pressure scientists and one representative member assigned by COMPRES. The Committee is responsible for making major recommendations to the Principal Investigators on the overall HPSynC project. The Steering Committee meets at least once per year, and its members are in contact frequently on an informal basis. The Committee provides advice on priorities, scientific direction, and service to the high pressure community.

3. Design Teams

Similar to GSECARS in early stage, we will establish Design Teams for each major direction. The design teams have the following responsibilities:

- Advise PIs on the scientific direction of the project in each area;
- Advise PIs on the technical design for each area;
- Help to organize workshops and other outreach activities to obtain input from the wider community;
- Maintain contacts with other synchrotron facilities and neutron facilities to keep abreast of new developments and coordinate design effort.

The four Design Teams at HPSynC are in areas of x-ray diffraction, x-ray spectroscopy, x-ray imaging, and time resolved experiments, with each area having a responsible staff at HPSynC. Each Design Team consists of 5-7 experts in both relevant scientific areas and synchrotron technical matters.

HPSynC Missions

1. Advancing Novel Techniques at Specialized Beamlines

Enormous capabilities of ultrahigh energy, submicron spatial resolution, sub-eV energy resolution, temporal structure, coherence, and myriad new spectroscopy, diffraction, scattering and imaging techniques are advancing rapidly at specialized beamlines. Many of these capabilities cannot be adapted or optimized at dedicated high-pressure beamlines, and the high-pressure community is missing some of the most exciting capabilities offered by the third-generation synchrotron source. As demonstrated by the aforementioned example, high-pressure experimentation can be developed at the specialized beamline. Collaborating with the beamline scientists, the HPSynC scientists will recommend all necessary modifications at the beamline, and make complementary modifications of the high P-T instrumentation. The CoPIs will also involve in the brainstorming and experimentation. Development carried out by the consorted effort of on-site HPSynC high-pressure experts as their primary mission will be far more efficient and reassuring than the previous mode by Mao and Lin as their side project. Other scientists in the HPSynC community may also initiate their development projects through collaboration with the HPSynC team and beamline scientists and make the development a community effort.

2. Integrating High P-T Instrumentation at Other Beamlines

With moderate investment and modifications, general diffraction and spectroscopy beamlines can accommodate high P-T experiments without interfering their standard programs. The HPSynC team will work with beamlines that are receptive to this idea and customize their sample stage, beamline focus, and detector geometry. The team will standardize the high P-T apparatus for maximum compatibility at different beamlines. The team will also consolidate the strength of various types of high-pressure apparatus, anvils, composite gaskets, sample assemblages, and sample loading procedures and to make them readily accessible to users. Coinciding with the arrival of radically new, superior, large anvils such as supported moissanite, ultrafine grain polycrystalline diamond, and giant single-crystal CVD diamond with imbedded circuits, the designs of multianvil, torroidal anvil, and diamond anvil presses may converge and advance to form the next generation high-pressure apparatus with much greater capabilities, efficiency, and user-friendliness

3. Increasing the High-Pressure Earth Materials Beam Time by >90 days/yr.

COMPRES has considered the option of solving the beam time shortage problem by joining HPCAT as a contributing member, but acquiring 60 days/yr beam time at HPCAT would have costed $3M initial construction investment and $400 K/yr operation expenses. The cost at GSECARS is similar.

HPSynC's integration of high P-T instrumentation at specialized and generalized beamlines will greatly improve the efficiency and success rate of high P-T experiments at these beamlines. The productivity of the high-pressure users will naturally improve their competitiveness in merit-based GUP beam time. Given the large number of beamlines at APS showing strong interests in high pressure projects, we estimate that will increase high-quality beam time for high-pressure Earth materials scientists by more than 90 days/yr (180 days/yr including non-geological high-pressure scientists), thus effectively solving the problem. In the initial stage before the overall beamline integrations are completed, HPSynC will work with HPCAT and APS GUP system to assure meeting the minimum number of 90 days/yr.

4. World-Leading High-Pressure User Laboratory

Successful high-pressure experiment critically depends upon the quality of sample preparation. HPSynC will establish a world-leading sample preparation laboratory that puts general users at equal ground as leading high-pressure center scientists who at present can prepare superior samples at home bases and carry the high-pressure cells to synchrotron facilities. In addition, the HPSynC staff will integrate other allied, non-synchrotron probes, such as Raman, optical spectroscopy and Mössbauer spectroscopy, and coordinate with nearby facility, such as the electron-microscopy center at ANL and the newly established Center for Nano-materials, to allow users to conduct truly comprehensive high P-T experiments.

5. Trainings, Workshops, and Community Outreach

The HPSynC staff will transfer newly acquired technology to the high-pressure community by topical workshops, and reach out to new researchers and students by regular training programs. They will also arrange consolidated purchasing and fabrications of special high-pressure cells, sample assemblages, anvils, tungsten carbide and c-BN seats, Be and Re gaskets, ruby spheres, and other key components and distribute to users on cost-sharing basis. Not only this will results in huge saving in cost and time, the tedious task of searching for unique sources and acquiring special components is among the top roadblocks that make high-pressure experiments difficult.

Scientific Focuses

HPSynC provides the technical platform for high-pressure Earth scientists to conduct cutting-edge research on their individual scientific agendas as shown in the following examples:

- High-pressure petrology and mineralogy –In studies of ultrahigh pressure metamorphic rocks or shocked meteorites, mineral inclusions in phenocrystals and in shocked veins often contain rich information on petrogenesis. The structure, texture, strain, chemistry, and exsolution of the micro-to-nanometer size inclusions are highly sensitive to the formation environment, thus revealing the physical and chemical process in great detail. Numerous new minerals have been discovered as micro-nano inclusions together with *in-situ* high *P-T* experiments that define the conditions of formation. Analogous to the trace-element geochemistry which has become the core of geochemistry, the emerging micro-nano mineralogy may take center stage in mineralogy-petrology with these new enabling synchrotron, high-energy, x-ray, nano-probes for analyzing experimental and natural specimen with 100 nm resolution.
- Deep Earth geochemistry –Recent studies of the Earth's deep interior present us with a rich array of large-scale processes and phenomena that are not fully understood. These range from the fate of deeply subducting slabs, the nature of the core-mantle boundary, the differentiation of elements to form the present day crust, mantle, and core, the distribution of trace elements, and volatile uptake and recycle in the Earth's history. With the new arsenal of synchrotron probes over the extended *P-T-X* regimes, we can now address questions that were not conceivable only five years ago. For instance, synchrotron x-ray emission spectroscopy identifies the high-spin to low-spin transition, one of the governing factors which determines Fe-Mg partitioning, the most important parameter in element differentiation throughout the mantle and core-mantle boundary; synchrotron x-ray absorption near-edge spectroscopy reveals electronic and oxidation state of transition elements in compressed solids and fluids; synchrotron x-ray Raman spectroscopy reveals the bonding nature of light elements that control the phase transitions, structure and partitioning of these elements; and synchrotron x-ray diffraction identifies new structures and phase assemblages in crystalline solids and melts.
- Mission to the core – The Earth's core plays a central role in the evolution and dynamic processes within the planet. However, answers to some of the most fundamental properties remain elusive (*e.g*. the temperature, chemical composition, crystalline phases, elasticity, magnetism, and formation of the core). Indeed, the "mission to the Earth's core" has united observational, theoretical, and experimental geophysics, and enriched each discipline through interactions and feedback. For instance, geophysical observations uncovered surprising inner core properties such as seismic anisotropy, super-rotation, and magnetism. These measurements include XRD under hydrostatic compression at inner core *P-T* conditions to yield robust, fundamental data on equations of state and crystallography. High energy-resolution, nuclear resonant x-ray spectroscopy provides information on the Mössbauer effect and magnetism, as well as the phonon density of states, bulk longitudinal and shear wave velocities, heat capacity, entropy, Debye temperature, and Grüneisen parameter.

High energy x-ray coupled with large sample volume at high P-T allows the investigation of liquid structure and viscosity of molten iron. The integrated approach for these closely related and complementary techniques will have a greater impact than the sum of individual studies.

- _Seismology and elasticity_ –Recent seismological studies have provided detailed descriptions of 3D tomographic images with regional and global discontinuities, heterogeneities, and anisotropy. Interpretations of these results require additional knowledge on the real materials that constitute the deep Earth and whose elastic and inelastic properties are drastically altered by its extreme *P-T* conditions. The long list of key properties include density and unit cell parameters, bulk modulus (K), shear modulus (G), single-crystal elasticity (c_{ij}) and velocities (V_P, V_{S1}, and V_{S2}), phonon density of state, and phonon dispersion. A series of breakthroughs in high-pressure physics and synchrotron technology has finally made it possible to propose a study of all these properties with newly-enabled, complementary, interactive techniques, including large volume ultrasonic interferometry, gigahertz interferometry, Brillouin scattering spectroscopy, radial x-ray diffraction, nuclear resonant inelastic x-ray scattering, phonon inelastic x-ray scattering, and x-ray thermal diffuse scattering. Essential ingredients of such development include sub-meV resolution x-ray monochromator and analyzers and >100 keV x-ray source that requires specialized beamlines but currently unavailable at general high-pressure beamlines such as HPCAT, GSECARS, or COMPRES facilities

Budget Justification

The proposed budget is very modest in view of the stated HPSynC goals: (1) revolutionizing the high-pressure synchrotron science, (2) acquiring 90 days/yr with superior capability, and (3) extensive community building and outreach. This is possible only because HPSynC is built on top of the existing APS beamlines, and is leveraging the enormous resource and expertise of APS which will absorb the cost of beamline improvement taking into consideration of the high-pressure science. Moreover, HPSynC benefits from the intangible wealth of high-pressure expertise from Geophysical Laboratory cumulated through CHiPR investment and beyond.

The $300K/yr is an infrastructure budget to start the HPSynC. This COMPRES fund will enable us to leverage additional funding for equipment and personnel from other sources. The eventual plan of the proposed HPSynC team consists of 3.5 scientists (including a part-time project manager), 2 engineers, and an office coordinator. In this COMPRES infrastructure proposal, one beamline scientist, 1 engineer, and an office coordinator (part-time at 0.25 level) are requested, and we are active seeking other sources for the full team. The APS management has already agreed to provide a full-time-employee beamline scientist position, laboratory and office space, and infrastructure support for HPSynC, and we have also got verbal commitments of $200K/yr for equipment and personnel matching from DOE, with both contingent to the success of the $300Kx3 yr COMPRES grant.

In the present budget, $8K/yr is requested for the scientist attending scientific meetings ($2K/yr) and for annual Steering Committee and design team meetings ($6K/yr). Modest budget (~$23K/yr) for materials and supplies is requested to start the HPSynC laboratories and for the cost of initiating high pressure experiments at specialized beamlines. We will submit separate proposals for funding major laboratory equipments.

Synergy with COMPRES

Although the proposed HPSynC budget will affect the overall COMPRES budget, we believe the effect is positive, i.e., the HPSynC will increase the total more than the sum of two individual proposals because HPSynC greatly enhances and complements three areas of the current COMPRES organization.

1. COMPRES focuses on facility and infrastructure of established technology; HPSynC merges new challenges together with facility, infrastructure and outreach.
2. COMPRES does not have direct access to APS, the most advanced facility for the COMPRES community. HPSynC is based at APS, and will provide significant amount of

beamtime (>90 days/yr) to the community.

3. HPSynC will reach and extend to Earth scientists who are not a part of the COMPRES structure. These include individual scientists who do not consider themselves as COMPRES members even a representative in their institution is, institutions that did not join COMPRES due to technical or legal issues, and areas of high-pressure research that have not been well represented by COMPRES.

Synergy with HPCAT and GSECARS

Both GSECARS and HPCAT are dedicated high pressure beamlines with major techniques developed and established successfully. HPSynC is a virtual beamline which is built on top of many existing APS beamlines (including HPCAT and GSECARS), and is leveraging the enormous resource and expertise of APS. HPSynC will develop many synchrotron techniques that have been advancing rapidly at specialized beamlines, but are unavailable at GSECARS and HPCAT. HPSynC will collaborate and coordinate with HPCAT and GSECARS, as well as all other APS beamlines to develop advanced techniques and sample preparation facilities. Based on the experience of HPCAT and GSECARS, HPSynC will establish and maintain a world-leading sample preparation laboratory that is open to all high pressure users at APS, including those of HPCAT and GSECARS.

Budget (in thousand US$)

HPSynC	**2006-2007**	**2007-2008**	**2008-2009**	**3 yr. Total**
Beamline Scientist (1)	$72,000	$74,500	$77,000	$223,500
Engineer (1)	$72,000	$74,500	$77,000	$223,500
Office Manager (0.25)	$10,000	$10,300	$10,600	$30,900
Total Salary	$154,000	$159,300	$164,600	$477,900
Fringe Benefit (28.5%)	$43,890	$45,401	$46,911	$136,202
Travel	$8,000	$8,000	$8,000	$24,000
Materials, Supplies, Anvils	$30,205	$23,395	$16,584	$70,184
Publications	$2,000	$2,000	$2,000	$6,000
Total Direct Cost	$238,095	$238,096	$238,095	$714,286
IC (26% TMDC)	$61,905	$61,905	$61,905	$185,714
Total (TDC + IC)	$300,000	$300,000	$300,000	$900,000

REFERENCES CITED

See proposal text sections for relevant references.

COMPRES Publications for 2002-2006 are referred to in Section A.9 and also included in total in Suppmentary Documents No. 4.

References associated with specific Community Facilities operations [B.1] and Infrastructure Development projects [B.1] are listed within each section of this proposal.