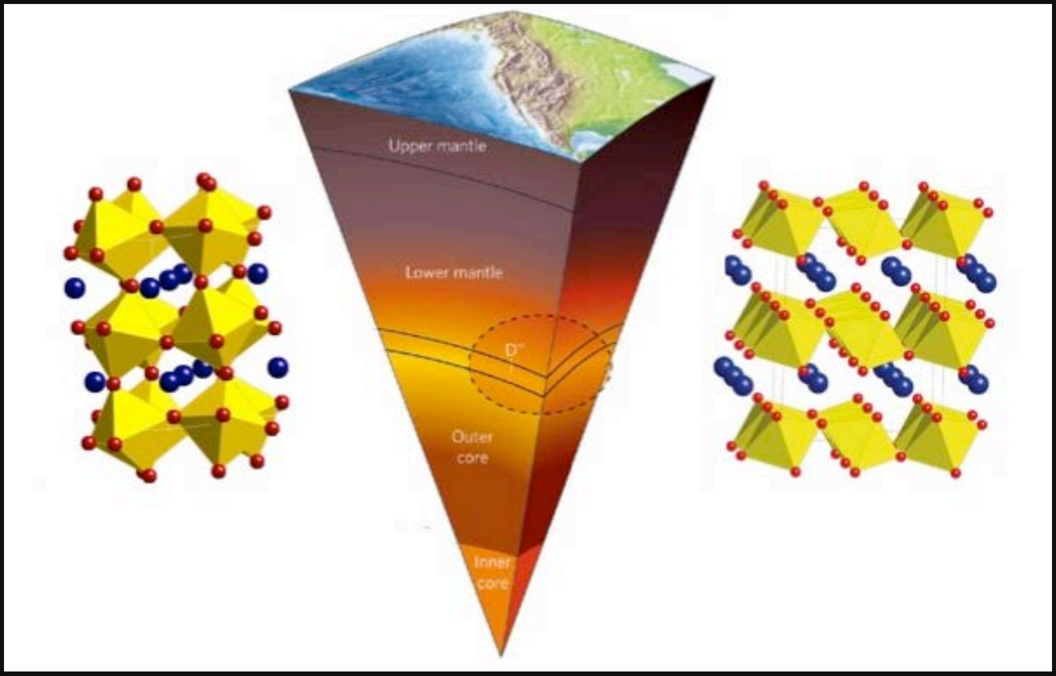
Understanding the Building Blocks of Our Planet: The Materials Science of Earth Processes



COMPRES:
The Consortium For Materials Properties Research in Earth Sciences

National Facilities and Infrastructure Development for High-Pressure Geosciences Research

Proposal to NSF 2012-2017

Volume I
Part A: Project Overview
Part B: Program Reports

Part B: Project Plans and Supplementary Information

Table of Contents

Acronyms used in this proposal

ADXD	Angular Dispersive X-ray Diffraction
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
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
D-DIA	Deformation DIA, A cubic anvil device for deformation studies
DMR	Division of Materials Research at NSF
DOE	Department of Energy
EAR	Division of Earth Sciences at NSF
EDX	Energy Dispersive X-ray analysis
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
HIX	Hard Inelastic Scattering Beamline, NSLS-II Project Beamline
HPCAT	High Pressure CAT at the APS
HXN	Hard X-ray Nanoprobe, NSLS-II Project Beamline
IF	Instrumentation and Facilities Program in EAR at NSF
IR	Infra-red
IXS	Inelastic Scattering Beamline, NSLS-II Project Beamline
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)
MIE	Major Item of Equipment (at NSLS II)
NRIXS	Nuclear Resonant Inelastic X-Ray Scattering
NSF	National Science Foundation
NSLS	National Synchrotron Light Source, a synchrotron facility at BNL
NSLS-II	National Synchrotron Light Source II, currently under construction
ORNL	Oak Ridge National Laboratory, TN
PE Cell	Paris-Edinburgh Cell
PRT	Participating Research Team, a group managing a beamline at the NSLS
PUP	Partner User Proposal
RDA	Rotational Drickamer Apparatus
SLAC	Stanford Linear Accelerator Center

SNS	Spallation Neutron Source, a neutron facility under construction at ORNL
Tcup	A type of two-stage multi-anvil system for use with synchrotron X-ray experiments.
XPD	X-Ray Powder Diffraction Beamline, NSLS-II Project Beamline
XAS	X-Ray Absorption Spectroscopy
XRD	X-Ray Diffraction

B.1: COMPRES Facilities

B.1.1 Synchrotron Facilities at the Advanced Light Source: A Proposal for Continued Support of the COMPRES West Coast Facilities

COMPRES PI: Q. Williams, UC Santa Cruz

Summary

We propose to, over the next five years, markedly augment the portfolio of the Earth Sciences high-pressure effort at the Lawrence Berkeley National Labs Advanced Light Source, while simultaneously maintaining the more standard capabilities that regularly serve our user community. Specifically, we plan to build on synergistic developments that we have made in radial diffraction, external heating and single-crystal diffraction to produce a facility with unusual and perhaps unique capabilities to accurately probe the properties of materials at pressures and temperatures spanning those present in the upper mantle, transition zone, and top of the lower mantle. At the same time, we plan to broaden our high-pressure effort to encompass a greater level of interaction between our primary diffraction (with laser-heating) beamline (beamline 12.2.2) and the highly complementary microdiffraction capabilities of the neighboring 12.3.2 beamline. Automation of pressure, temperature and spatial mapping as well as ease of use for better efficiency and remote operation will be an operational theme for beamline use. Our in-house sample preparation facilities will be completed in the first year of the proposed project, with a state-of-the-art laser milling set-up and a gas-loading apparatus. To accomplish these goals, and continue to provide state-of-the-art service to users, we request continued support for two beamline scientists, as well as funding for expendable materials and prospectively software development.

Present High-Pressure Facilities and Operations at the Advanced Light Source

Under the aegis of COMPRES, the Advanced Light Source has provided key infrastructure for high-pressure Earth Sciences research for a broad suite of national and international investigators. While our "core" user-base is largely from the Western U.S., a region with a long-standing and vibrant high-pressure community within the Earth Sciences, the ALS capabilities have attracted, in our particular areas of focus, a global clientele. Over the last interval of funding, the ALS COMPRES effort has concentrated on (1) improving our high-pressure laser-heating set-up so that it is a user-friendly, state-of-the-art synchrotron laser-heating apparatus (a process which is just reaching fruition); (2) expanding on our considerable capabilities in radial diffraction (with applications to the high-pressure rheology of materials) which have been one of the long-standing hallmarks of the ALS high-pressure enterprise; and (3) making significant advances in externally-heated diamond anvil cell work—an area in which we have recently pioneered new extremes of temperature. Each of these efforts is centered around high-pressure beamline 12.2.2. At the same time, we have improved our sample preparation infrastructure, as well as our management structure and our processes for implementing user suggestions and making improvements to our systems. Our net goal is to provide a user-friendly facility that can allow users to accomplish a broad suite of measurements, from comparatively routine high-pressure diffraction measurements, to more challenging areas in which the interests of our user base coupled with our on-site expertise have led us to focus.

COMPRES funding has allowed high-pressure Earth Sciences synchrotron work on the West Coast to flourish.

Indeed, COMPRES has provided the linchpin support that has leveraged not only beamtime, but also extremely significant resources from the Advanced Light Source itself. In particular, the ALS has provided both staffing and extensive infrastructure in addition to that provided by COMPRES for high-pressure beamline 12.2.2: senior beamline manager Alastair MacDowell is assigned to 12.2.2 (33% time), ands is technical support person Jason Knight (100%). Each provide trouble-shooting expertise and user support beyond those supplied by the COMPRES technical support. Indeed, the overarching expertise of MacDowell (who designed the original beamline set-up of 12.2.2, as well as multiple other beamlines at the ALS) and the technical experience of Knight provide an enormously valuable in-kind match for the COMPRES employees. Selva Venilla Raju, who has been employed by COMPRES as a Researcher, was also funded for 8.2 months in 2010-2011 by the Advanced Light Source to continue her work on external heating. Additionally, Simon Clark (ALS beamline scientist) is charged by the ALS with support for materials science-oriented users at beamline 12.2.2.

With respect to infrastructure, the ALS has, over the last year, purchased a gas-loading apparatus in the well-known GSECARS design for the high-pressure sample preparation laboratory ($275 K) from the University of Chicago machine shop (delivery date = 12/11), and provided an extended loan of a Bruker PT200 CCD detector (~200 K) to the 12.2.2 high-pressure beamline. These capital commitments are in addition to the many day-to-day supplies for the beamline and users (replacement parts, optics, spare cells) that are funded by the ALS at the approximately 80 K/yr level. In short, the West Coast COMPRES enterprise has engaged in a highly successful partnership with the ALS: one that has produced both high levels of staffing and equipment infrastructure, each of which are directed towards generating productive user experiences.

COMPRES funding has leveraged not only beamtime, but also substantial staffing and infrastructure from the ALS.

The assignment of beamtime at 12.2.2 is conducted through two avenues: (1) the COMPRES Approved Program (AP: guaranteed 35% of the beamtime); and (2) the DOE-mandated General User program. COMPRES-affiliated users can access both routes—time is awarded in each category through ratings derived from competitive reviews conducted by the ALS Proposal Study Panel. COMPRES users typically receive between 50 and 70% of the available beamtime at 12.2.2. For example, in the most recently completed July-Dec. 2010 cycle, COMPRES users received 150 of the 252 allocated shifts (~60%) of shifts in the July-Dec. 2010 cycle (see Appendices 3 and 4). The typical oversubscription rate (the ratio of time awarded to time requested) for beamtime at 12.2.2 is ~2.6. From an administration standpoint, there is a clear recognition within the ALS management that the COMPRES AP is unique among the AP's at the ALS, as (in contrast to all other AP's), beamtime for the COMPRES PI (or their affiliates) is allocated through the proposal system on a competitive basis. That is, there is no *quid pro quo* of AP beamtime for the COMPRES PI: this feature is critical for our recognition within the ALS as a broad-based community enterprise, and our associated leveraging of ALS resources.

Traditionally, COMPRES has allocated 2 beamline scientists at the Advanced Light Source (due to carry-forward from unfilled positions or Infrastructure Development Grants, there have been 3 employed for much of 2009-2011); given the high-demand from COMPRES users for assistance and custom experimental set-ups (particularly for radial diffraction and external heating, as described in the next section), this allocation represents a minimum for viability of the COMPRES 12.2.2 enterprise. When 3 beamline scientists have been available, there has been sufficient manpower to aggressively pursue new initiatives (such as pushing the limits of high-pressure external heating). Nevertheless, recognizing budgetary constraints, our budget is oriented primarily towards salaries for 2 beamline scientists, with some funding for expendables and possible software development, and for travel for beamline scientists to both meetings and to other facilities to observe and share best practices.

Personnel are the key to the success of the high-pressure geosciences community at the ALS.

Operation Plan and Priorities for the Next Five Years

This is an extremely exciting time in high-pressure science at the ALS. Dramatic new temperature-pressure records have been established by our external heating enterprise; exciting new science has emerged on the rheology of deep Earth materials from our radial diffraction capabilities; and a large influx of new infrastructure has come, or will shortly come on-line (a new x-ray detector on extended loan, a gas-loading apparatus on order, and a laser-mill recently arrived on site). And, the publication rate from beamline 12.2.2 has steadily improved through time (Figure 1). In short, the overarching goal of the ALS high-pressure effort to provide state-of-the-art facilities coupled with superb support for the user community is moving forward rapidly.

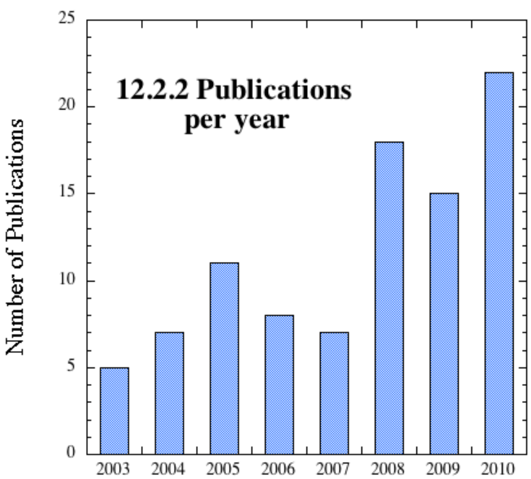


Figure 1. Number of refereed publications per year associated with high-pressure beamline 12.2.2 (as reported in the ALS publication database).

The ALS COMPRES enterprise fully recognizes that its mandate is to provide service to users for both relatively routine experiments (usually 300 K x-ray diffraction of statically compressed samples, either with-or-without quenching from high-temperatures) and substantially more challenging state-of-the-art experiments. The former capabilities are well-established, and we naturally plan to ensure that users with these needs continue to be supported: because they conduct comparatively straightforward experiments, the rates and numbers of publications of users with relatively routine needs are somewhat higher than those who conduct higher-risk-higher-reward experiments. As such, the benefit-to-effort-expended ratio is high for such studies—and at times, such experiments produce results defined as high-profile by DOE criteria (such as Friedrich et al., Novel Rhenium Nitrides, Physical Review Letters, 2010), or with major Earth Sciences implications (such as Wicks et al., Very Low Sound Velocities in (Mg,Fe)O: Implications for the Core-Mantle Region, Geophysical Research Letters, 2010). With this commitment to "routine" studies over the course of COMPRES-III being taken as a given, we will primarily focus our discussion on five areas of achievement and development which we plan to expand and augment over the next five years. These are: (1) continuing our development and upgrade work with our laser-heating system, with the net goal of making it among the most user-friendly and productive simultaneous high-pressure, high-temperature diffraction set-ups in the Earth Sciences; (2) improving and expanding our radial diffraction capabilities, which have already produced extremely high-profile results on the rheologic behavior of deep mantle minerals; (3) pushing the limits of externally-heated diffraction studies—an area in which we have made considerable progress over the last year; (4) establishing the capability for simultaneous high-pressure and high-temperature single-crystal x-ray diffraction studies; and (5) enhancing our interactions with highly complementary beamlines at the Advanced Light Source, and particularly the adjoining 12.3.2 microdiffraction beamline, which can characterize the structural and chemical differences in quenched samples at the ~1 micron or less length-scale—a capability with extensive applications in characterizing the multi-phase run products of chemically and mineralogically complex geologic/geophysical samples. Naturally, we believe that there is a strong likelihood that our detailed directions might shift in response to user interests or unanticipated advances over the next several years: such reactivity in response to serendipitous developments is, we believe, a key aspect of our past successes.

Laser Heating

The importance of establishing the ALS as a reliable and world-class high-pressure laser-heating facility has been forcefully emphasized on behalf of our community by both a CALIPSO (California High Pressure Observatory) external review committee report of January, 2007, and subsequently by a Report on the COMPRES high-pressure enterprise at the ALS conducted by the COMPRES Facilities Committee of January, 2010. Hence, the primary focus of the ALS and COMPRES staff over the last year has been on improving the laser-heating setup. Our general approach has been to (1) where possible, simplify the existing system; (2) rigorously quantify the optical and overall system response and performance; (3) develop new systems off-line, such as new beam-shaping optics and a 4-color pyrometry system; (4) where possible, simulate the system performance using standard optical codes, such as ZEMAX; and (5) improve computer control and diagnostics to facilitate user operation.

ALS staff has, in conjunction with COMPRES personnel, conducted this sequence of ongoing tasks oriented towards making the system consistently reliable and user-friendly, improving the characterization and benchmarking of the system, and improving the optics and software of the system so that (for example) temperature gradients are reduced and characterized. The rationale for this focus on accurate and user-friendly laser-heating is straightforward: such capabilities are in high demand in the community, as nearly all of our experimental constraints on the high-pressure and temperature behavior of the planet below ~1000 km depth are derived from such laser-heated diamond anvil cell experiments. Our laser-heating system has produced some outstanding Earth Sciences results (e.g., Lord et al., The FeSi phase diagram to 150 GPa, JGR, 2010; Walker et al., X-ray absorption contrast images of binary chemical reactions, Chemical Geology, 2009), but maintaining this system at the state-of-the-art while regularly improving it in response to user suggestions is an ongoing, time-intensive process that we plan to continue over the next five years.

Radial Diffraction

The ability to conduct radial diffraction (diffraction on samples perpendicular to the access of force) in the diamond anvil cell has revolutionized the study of deformation mechanisms in minerals at high pressures. How differential stress and texture evolve within compressed (or, in some instances, phase transforming) samples has been an area of primary focus at 12.2.2 since 2006. Indeed, the diffraction setup at beamline 12.2.2 was designed to be sufficiently flexible that the experimental configuration can be rapidly shifted between axial diffraction and radial diffraction. Research at the ALS has spanned the gamut of major lower mantle mineral phases, with recent results on the post-perovskite structure of (Mg,Fe)SiO_3 providing results that provide fundamental constraints on the origin of seismic anisotropy in this geophysically crucial region (e.g., Miyagi et al., Slip systems in $MgSiO_3$ post-perovskite: Implications for D" anisotropy, Science, 2010).

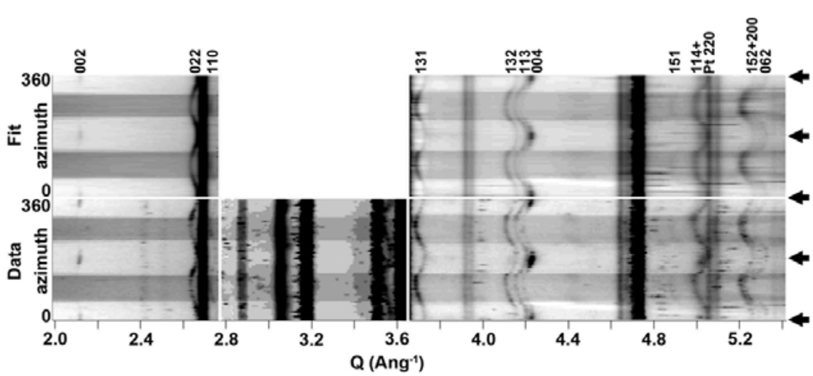


Figure 2. Radial x-ray diffraction results on $MgSiO_3$ post-perovskite at a pressure of 185 GPa following laser-heating; bottom panel is the data, while the top panel shows the Rietveld fit to the data from the textured, polycrystalline sample (the region from 2.8 to 3.6 Å is

excluded from the calculation due to the presence of gasket lines).

To date, most of our radial diffraction data has been taken either on simple 300-K compression, post-laser-heating, or with modest degrees of external heating. We plan over the course of COMPRES-III to enable our users to conduct in situ experiments at simultaneous high pressures and temperatures, using both the external heating techniques we have been developing (see following section) and under laser-heating. Indeed, because of the crucial role of temperature gradients in potentially shifting deformation mechanisms of materials, we believe that a combined laser-heating/external heating approach may be optimal for both generating Earth-interior-relevant extremes of pressure- and temperature with a minimization of thermal gradients.

Resistive (External) Heating

We have recently made a very major advance in capabilities and techniques on which we plan to expand over the next funding cycle of COMPRES. Briefly, our external heating effort (with a furnace mounted around the diamond anvils) achieved dramatic success with a modified Liermann-type radial diffraction diamond anvil cell (conducted in conjunction with the Wenk group at UCB), achieving record-setting conditions for such experiments: 40 GPa and 2000 K in an externally resistively heated cell (Figures 3 and 4). It is likely that these conditions can be improved upon, with design improvements in progress; further work on our resistance heating technology was funded in 2011 COMPRES Infrastructure Development Proposal (titles HEETDAC; see end of this section). Notably, these conditions span those within planet Earth to depths corresponding to those at the top of the lower mantle (Fig. 4). In essence, our results indicate that the diamond anvil cell may be able to access, using external heating (and thus without the temperature gradients endemic to laser-heated experiments) the pressure-temperature range currently accessed by large-volume presses. The prospect of being able to utilize the optical transparency of diamond to probe materials in a controlled environment within this pressure-temperature range opens up a broad suite of in situ probing of Earth materials as they undergo reactions, phase transitions, and even partial melting.

Thus, we view external resistive heating as a valuable and absolutely cutting-edge addition to the capabilities at the ALS—based solely on initial brief informal communications of these results, users are already expressing strong interest in these new capabilities. In addition to advancing and making more routine our external heating capabilities, other development possibilities associated with such heating will also be probed during the period of funding. For example, the configuration at beamline 12.2.2 is particularly amenable to laser-heating within a resistively-heated cell because of the relative geometries of the laser beam, X-ray and DAC axis in our radial diffraction geometry (Miyagi et al., In-situ phase transformation and deformation of iron at high pressure and temperature, J. Appl. Phys., 2008). Substantial prospects hence exist to both access higher temperature conditions, but also (and perhaps more importantly) to access higher temperatures with markedly minimized temperature gradients, and larger laser-heated spots—a configuration that could prove particularly valuable for (for example) accurate high-pressure and temperature melting studies, or characterization of solid-solid phase boundaries.

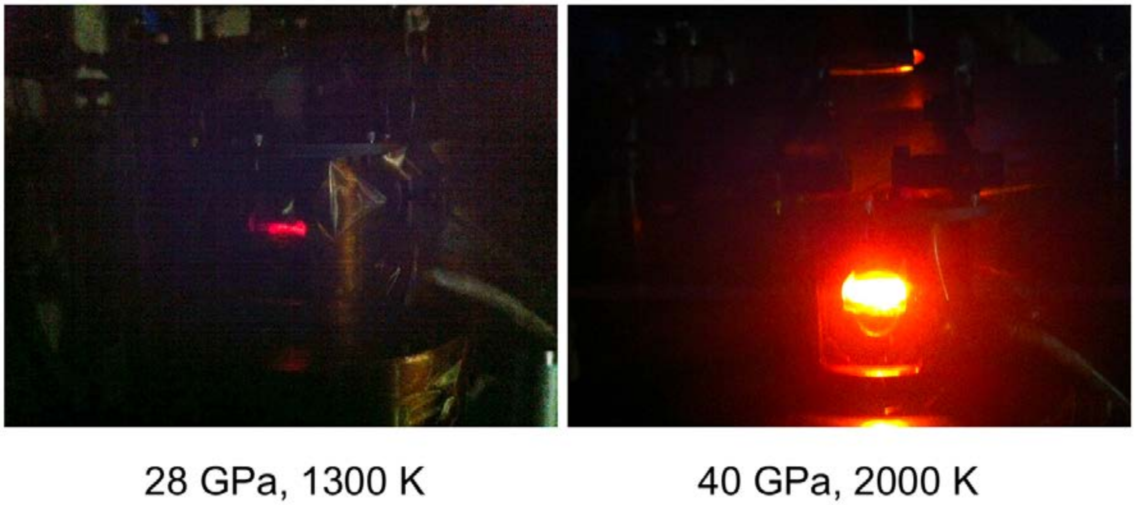


Figure 3. Images of the modified Liermann cell at the ALS in radial diffraction mode during a recent high pressure, high temperature experiment. Temperatures were calibrated using Pt-Rh thermocouples, and pressures from the equation of state of iron. Image from Jane Kanitpanyacharoen (UCB).

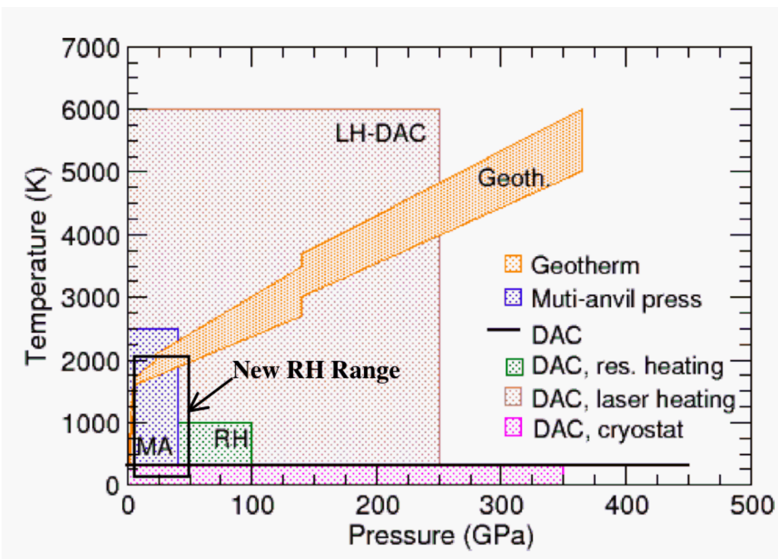


Figure 4. Pressure-temperature ranges of different experimental techniques; green box denotes a (slightly conservative) estimate of prior resistive heating capabilities; the black box denotes the range recently achieved at the ALS (Figure modified from S. Sinogeikin's modification of G. Shen's figure).

In the longer term (beyond the single year timeframe), we expect that such external heating will be a prominent area of emphasis for 12.2.2—and the advantages presented by the prospect of simultaneous laser-heating and external resistive heating may also become important for experiments at the ALS. Indeed, the current experimental geometry is such that simultaneous laser-heating and resistive heating could be relatively straightforward to achieve, and the advantages of such an approach (including smaller temperature gradients, larger laser-heated spots and greater hot spot stabilities) are likely to provide the motivation necessary to make such experiments feasible (if not entirely routine) at the ALS.

Single-Crystal Diffraction

A collaboration of S. Clark (ALS), P. Dera (GSECARS) and O. Tschauner (UNLV) has recently successfully implemented high-pressure single crystal diffraction capabilities at 12.2.2. Significant commissioning time has been dedicated to this project over the last two beamtime cycles, and preliminary reports are that excellent benchmarking data (on single-crystal quartz) has been collected. The GSECARS custom software that is designed to utilize a standard 2-D detector to collect single crystal diffraction data has been installed at the ALS, and is fully operational. Presuming that this effort continues to be successful (no fundamental impediments are apparent), the prospect (particularly when the new gas-loading system is installed at the end of 2011) is that high-pressure single-crystal data should be able to be collected to pressures spanning those present within the lower mantle of the Earth. Indeed, our anticipation is that the combination of this effort with our expertise in external heating will allow single-crystal diffraction experiments over an unprecedented pressure and temperature range. Indeed, we believe that the ALS high-pressure beamline should have the capability to not only conduct extremely accurate thermal expansion measurements at high pressure, but also provide the thermodynamic cross-check of accurate temperature dependences of bulk moduli at pressure. Such capabilities will also allow accurate studies of highly temperature-dependent phenomena of likely deep Earth importance, such as shifts in order/disorder, at high pressures. Hence, we fully expect that this new capability will attract additional COMPRES users to the ALS over the next five years, and we anticipate that the COMPRES employees will substantially engage with this effort over the next cycle of COMPRES.

Synergistic Interactions with other Beamlines

An area that has been emphasized by multiple prominent users of the ALS is the proximity and complementary character of 12.2.2 and 12.3.2—the microdiffraction beamline run by Martin Kunz (the former COMPRES staff member at 12.2.2) and Nobumichi Tamura. The scientific motivations for this interaction is simple: synthesis and high P/T characterization of samples can be conducted at 12.2.2, followed by detailed chemical and structural imaging of the quench products at 12.3.2. Indeed, results of a joint study at 12.2.2 and 12.3.2 generated one of our high-profile papers of the last year (Friedrich et al., PRL, 2010, on rhenium nitrides), and the prospect for future studies along these lines has the prospect of providing an additional valuable scientific niche for the ALS. The organization and time-scheduling procedures at the ALS render getting joint/simultaneous time on two beamlines quite feasible (with the natural caveat that the beamtime proposal is rated highly), and we hence view future closer collaborations/user interactions with 12.3.2 (and perhaps other stations around the ring) as a natural area of expansion over the next several years. These synergistic capabilities hold the prospect for establishing a particular niche for the ALS within the high-pressure geosciences community: synthesis and high P/T characterization of experimental charges, followed by immediate characterization of the compositions and structures of individual grains within the sample.

Automation

The nature of current high pressure experiments is very "hands on," which requires near constant attendance and monitoring by the user at the beamline during an experiment. Automation of many of the tasks is practical with instrumental and software development. The aim would be to systematically map out the diffraction patterns with pressure, temperature and spatial position on the sample in a fully automated manner. The data collection rate would be significantly increased leading to more experiments, more complicated experiments, and more user throughput. Such automation would also allow users to get some sleep during their runs, which helps improve experimental decision making. The end stations at the ALS are operated in the LabView environment with custom code written and managed by professional systems programmers. The code is modular and allows for development in different areas as resources and demand require. Expanding the current code to be more user friendly and involving automation would likely require about ten weeks of a programming effort that can be supplied from the LBNL software engineering pool: some programming can be accomplished by our technical staff, but a portion of our supply money could be utilized to purchase high-level programming support (the typical rate for the LBNL software engineering pool is ~16 K/month: even a few weeks of programming support (derived from our supply budget) over the course of the COMPRES III renewal would markedly enhance our automated capabilities.

On-line data analysis

A number of users do not carry out data analysis as the experiment proceeds. This can lead to less than optimal choices for the preferred experimental direction. Although data analysis of powder patterns is relatively straightforward, it could be carried out easier and faster and the various software packages available can be made to talk to each other if suitable software-wrappers are made. We note that the beginnings of such an approach has already been carried out by one of the current COMPRES scientists at the ALS (J. Yan: e.g., Yan, et al., The development of an automated data analysis system for powder diffraction data collected using an area detector, High Pressure Research, 2008). One of our focuses over the course of COMPRES-III will be to build on these earlier achievements to produce a suitable streamlined data analysis package that will produce data analysis in near real time, thus markedly enhancing both user productivity and their experience at the facility.

Remote Operation

Our motivations with respect to automation and remote operation are partially guided by the extensive experience of the ALS Protein Crystallography beamlines. For these beamlines, automation, sample mail-in service and remote operation on behalf of users has, perhaps counter-intuitively, led to a greater involvement by PI's in the actual experiment. This clearly leads to better decisions being made during the experiment —in lieu of experimental decisions being made by an on-site (and perhaps fatigued) student or post-doc, a PI can, in consultation with their group, guide on-line the progress of the experiment.. Remote control software is now quite advanced, but some development is required to match bandwidths, screen sizes and to have relevant data relayed to the user. The software development associated with instituting a remote operation component is also estimated as taking about 3 months of a programming effort that can be supplied from the LBNL software engineering pool. If mail-in service is ultimately instituted, then the COMPRES personnel would be required to mount the samples. However, students or post-docs attending the beamline can be expected to mount samples and then work with their supervisor as the experiment proceeds. This type of remote operation, with complete interaction between on-site and off-site users, represents the likely future of how user-oriented synchrotron radiation facilities will operate; indeed, it is the manner in which Protein Crystallography beamlines already operate, and we view it as a probable direction for extensive development at the ALS by both our employees and prospectively software engineers at LBNL over the course of COMPRES-III.

Management

Significant changes in the management of the COMPRES ALS enterprise occurred on June 1, 2010 (the start of year 4 of COMPRES II). The changes were induced by a Report by the COMPRES Facilities Committee on the COMPRES-funded effort at the ALS, produced in conjunction with a site visit in December, 2009, and which included extensive interviews with users, staff and the PI's associated with the high-pressure effort at the ALS. The Report recommended that the COMPRES management of the beamline be changed; and, ALS management recognized in turn the need for a change in the configuration of ALS staffing at the beamline as well. Thus, the PI-ship was shifted from R. Jeanloz (UCB) and S. Clark (ALS/UCB) to Q. Williams (UCSC), and the COMPRES employees were transferred from employment at UCB to UCSC.

The primary marching orders from the Facilities Committee Report were to redouble and refocus efforts to ensure that the ALS laser-heating system was reliable, robust and user-friendly; to truncate a nascent, off-line high-pressure laser spectroscopy program that had been pulling effort away from the primary beamline; and to improve the communications and interactions between the COMPRES and ALS stakeholders. The goals with respect to the spectroscopy program were almost immediately accomplished, with a Brillouin spectroscopy set-up being moved (at considerable expense to the ALS) to a lab at UC Berkeley. Improvements in the laser-heating system have been the focus of most of 2010-2011; changes in optics, in the mechanical robustness of the system, and in the control and measurement software have all been instituted. Protocols have also been established to prevent and identify misalignments. Finally, weekly all-hands meetings were established at which primary and secondary responsibilities for different user groups are assigned, issues arising at the beamline and a post-mortem on recent user time are discussed, and an ongoing task list is updated at these meetings, which contains specific assignments spanning from the immediate to the relatively long-term (couple of months) for each beamline-affiliate. The prioritization and time frames of different tasks (and introduction of new ones) is discussed and resolved at each meeting. Ultimately, all principals agree on these assignments, priorities and tasks. Figure 4 illustrates the present management structure, which we believe is working well.

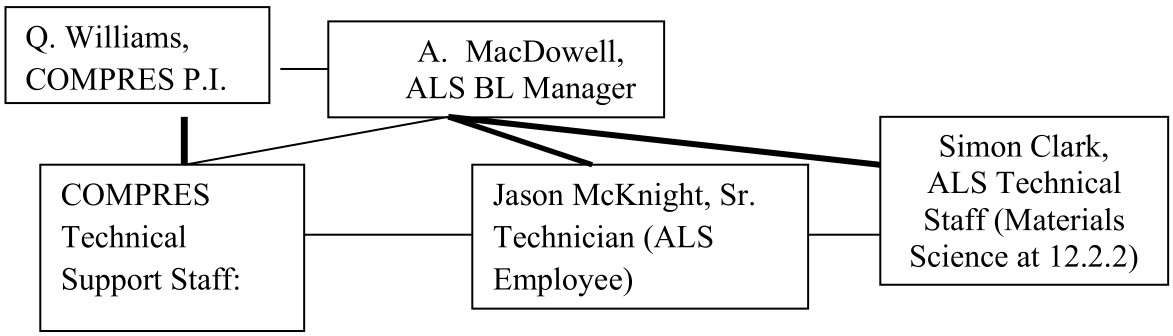


Figure 4. Representation of beamline 12.2.2 management structure; lower 3 boxes are user-support centered, and bold lines represent direct supervisory roles. Light lines represent consultative and advisory roles. User recommendations and difficulties are conveyed to the top level of the structure, and weekly all-hands beamline staff meetings are used to establish corresponding weekly individual and group priorities,with timelines for task completions.

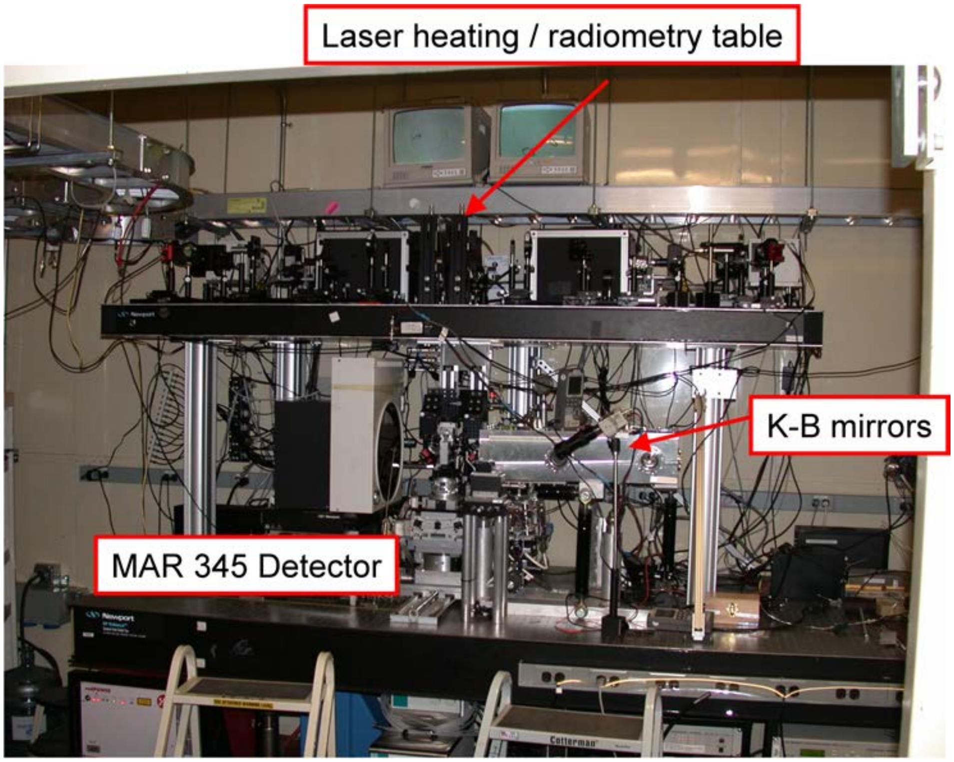
Publications

For the time period 2006 to July 2011, 88 publications and graduate student theses have resulted from COMPRES support of beamline 12.2.2 at the ALS. These are listed in the complete list of COMPRES publications submitted as a supplementary document to this proposal.

Appendix 1

Brief Summary of ALS COMPRES Facilities

Beamline 12.2.2 (Axial and radial diffraction and imaging with laser and external heating). The beamline is on one of the superbend sources at the Advanced Light Source, and is a hard x-ray (8-40 keV) focused synchrotron beamline. The two endstations of this beamline are oriented towards single-crystal x-ray diffraction measurements (endstation 1) and the smaller focal spot (ca. 10 microns x 10 microns) endstation 2, which is equipped with a double-sided laser-heating and spectroradiometry setup and an in-hutch ruby fluorescence system for pressure characterization. Either a MAR345 image-plate system or a Bruker P200 CCD can be deployed for x-ray detection. Either axial or radial diffraction studies can be carried out on endstation 2.

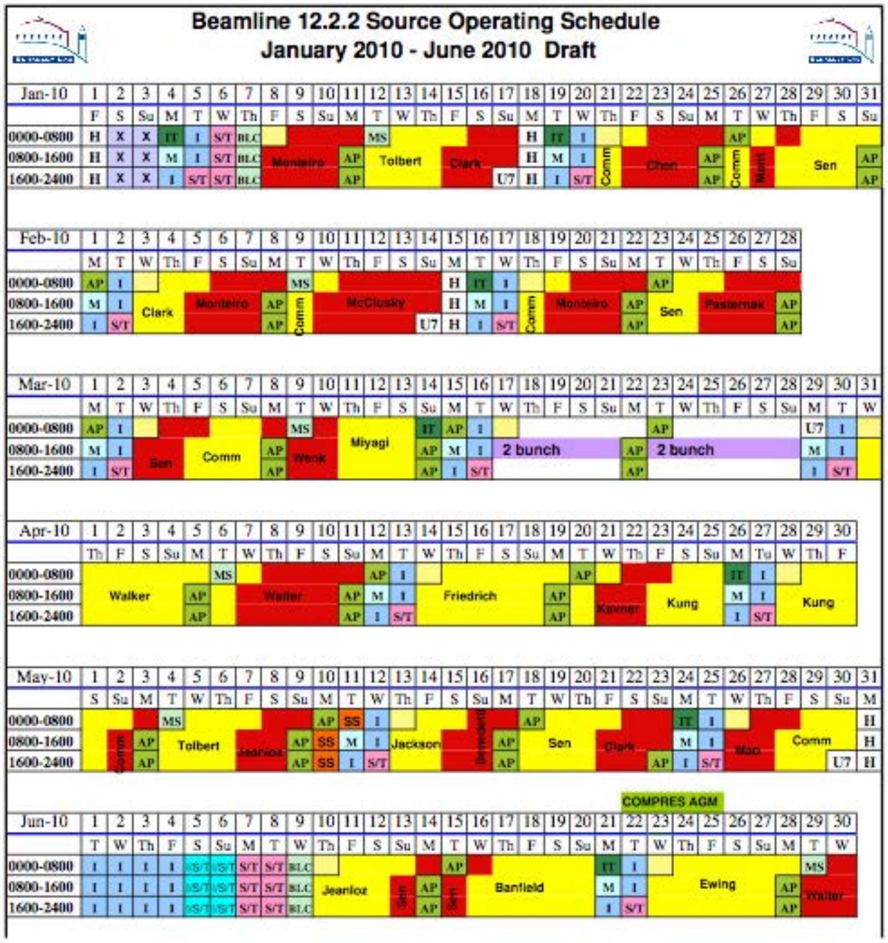


Beamline 12.2.2 at the ALS; samples are typically positioned to the right of the MAR detector, and the laser heating system primarily occupies the upper table, with the beam being steered into the sample area through holes in the upper table.

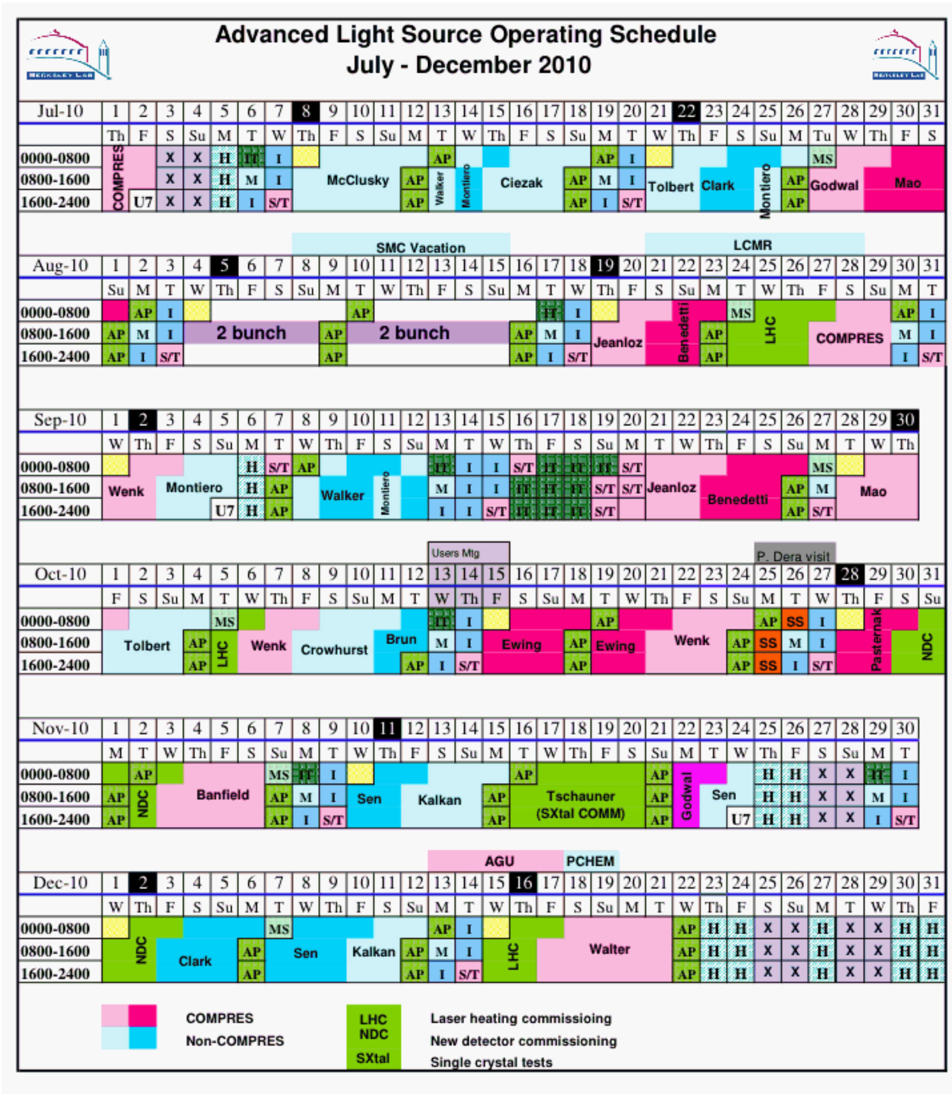
High Pressure Sample Preparation Laboratory: A fully equipped high-pressure sample preparation laboratory is located ~15 meters from the beamline. This laboratory is equipped with microscopes, micro-tools, an Oxford Instruments computer-controlled laser-miller, cryogenic loading facilities, and a customized station for preparing external heating apparatuses. A high-pressure gas-loading system (on the GSECARS design) is on order, and is scheduled to be installed in November, 2011. The ALS also keeps a moderate number of diamond anvil cells (~8) available for user usage, should the cells that the users bring fail or encounter logistic difficulties.

Appendix 2. Beamline Schedules

12.2.2 2010 Cycle 1.



12.2.2 2010 Cycle 2.



In the July-Dec. schedule, COMPRES time is (with the exception of Tschauner's single-crystal commissioning time and D. Walker's Sept. time) shown in pink and red.

Appendix 3. Representative Beamtime Requests for a Cycle (July-Dec. 2010)

ALS Shifts Allocation (July 2010 – December 2010)

C	GU	Investigator	Title	BL	Shf Req	Alternate Beamlines	Other Beamlines
12		Wenk, H	Simultaneous high-pressure and ?temperature investigation of texture development in polycrystalline $MgSiO_3$ perovskite	12.2.2	12		
	15	Kalkan, B	Is there really a polyamorphic phase transition in SnI_4?	12.2.2	15		
27		Walter, M	Water in the Deep Earth	12.2.2	15		
45		Ewing, R	Phase Transitions Induced by Simultaneous Exposure to Relativistic Ion Beams and High Pressure	12.2.2	18		
	24	Ciezak, J	High-Pressure Crystallographic Analysis of Nitrogen/hydrogen mixtures	12.2.2	9		
	42	Tolbert, S	High pressure studies of ultra-incompressible, superhard metal borides	12.2.2	18		
	51	Crowhurst, J	In situ x-ray diffraction study of the decomposition products of a high nitrogen content precursor	12.2.2	9		
	72	Sen, S	Pressure and temperature induced structural and topological changes in molecular chalcogenide glasses	12.2.2	20	21	
54		Wenk, H	Investigation of Texture Memory and Variant Selection during Phase Transformations in Uranium	12.2.2	9		
72		Paz-Pasternak, M	Search for a Mott-Hubbard transition in NiO, NiI_2 and $NiCl_2$	12.2.2	21	18	
	90	Weinberger, M	In situ high pressure X-ray diffraction of new green energetic materials	12.2.2	18		
90		Walker, D	X-ray Absorption Contrast Images of Binary Chemical Reactions	12.2.2	21	18	
105		Mao, W	Structural studies of 3d transition metal compounds at high pressure	12.2.2	48	~~18~~ 15	
120		Benedetti, L	Melting curve of graphite from ambient pressure to ten gigapascals	12.2.2	18	15	
135		Jeanloz, R	Platinum and iron phase boundaries in the laser heated diamond anvil cell	12.2.2	27	15	
	105	Friedrich, A	Formation and structure of binary transition metal carbides, -nitrides, and -borides at extreme-(P,T) conditions	12.2.2	18	15	
147		Banfield, J	Determining interfacial free energy of iron oxyhydroxide nanoparticles in various surface environments using synchrotron X-ray diffraction	12.2.2	21	Alternate Beamline 1: 11.3.1 for Beamline: 12.2.2 Shifts Req: 21 Entire Prog: 12	
		McCluskey, M	X-ray diffraction of ZnO, MgZnO, and CdZnO under pressure	12.2.2	12		
		Godwal, B	High-Pressure Elasticity and Stability of Osmium: An Analog for Iron in Earth's Core	12.2.2	12	Alternate Beamline 1: 9.0.1 Shifts for Cycle: 12 Shifts for Entire Program:	

ALS Shifts Allocation (July 2010 – December 2010)

	Investigator	Title	BL	Shf Req	Alternate Beamlines	Other Beamlines
	Godwal, B	Angle Dispersive X-ray Diffraction Studies on GaCMn3 under High Pressure	12.2.2	12		
	Monteiro, P	Effect of pressure on the structure of calcium silicate hydrates	12.2.2	25		
	Godwal, B	High pressure high temperature Angle dispersive X-ray diffraction (ADXRD) studies on Intermetallic Compound Auln2.	12.2.2	9		
	Raju, S	Study of thermal equation of state of Dilute magnetic semiconductors	12.2.2	21		
	Kung, J	Searching perovskite to post perovskite transition in (Ca,Sr)SnO3 at high pressure and high temperature	12.2.2	18		
	Chen, B	Elasticity and Structural Stability of Natural and Commercial Silicate Glasses at High Pressures	12.2.2	12		
	Gleason, A	The fate of iron-hydroxides at very high densities; structural and spectroscopic studies of goethite (FeOOH) and jarosite KFe3(OH)6(SO4)2.	12.2.2	21		
	Geballe, Z	Wet vs. dry post-spinel transition in resistive and laser heated diamond anvil cells	12.2.2	15	Alternate Beamline 1: 1.4.4 Shifts for Cycle: 9 Shifts for Entire Program:	1.4.3
	Lipinska-Kalita, K	Hybrid Glass*]Nanocrystal Composites at High*]Pressures	12.2.2	32		
	Crowhurst, J	In situ x-ray diffraction study of novel uranium compounds formed under extreme conditions of pressure and temperature	12.2.2	3		
	Braun, A	Influence of mechanical pressure on the proton conductivity of the Y-substituted barium zirconate proton conductor at elevated temperatures	12.2.2	15		
	Monteiro, P	Effect of pressure on the crystal structure of organic/inorganic Calcium Silicate Hydrates nanocomposites	12.2.2	8		
	Godwal, B	High pressure high temperature Angle dispersive X-ray diffraction (ADXRD) studies on Intermetallic Compound Auln2.	12.2.2	12	Alternate Beamline 1: 9.0.1 Shifts for Cycle: 12 Shifts for Entire Program:	
	Lee, K	Solidus Determination of Hydrous Pyrolite at Lower Mantle Conditions	12.2.2	16		1.4.3
	Godwal, B	High Pressure High Temperature X-ray Diffraction and melting Curve Studies on Osmium	12.2.2	9		
	Manghnani, M	Melting relationships and density of Fe-Ni based alloys and melts at high P-T: Implications for the Earth's core	12.2.2	24		
	Chan, S	Study of the size effect on bulk modulus of cerium oxide nanoparticles by X-ray diffraction under high pressure	12.2.2	18		
	Lipinska-Kalita, K	Compressibility and Structure of Ion Conductive Ceramics from the NaSICON Family for Low Cost Biodiesel Production	12.2.2	12		
	Yan, J	The composition effect of Gillespite on phase transition	12.2.2	9		
	Gleason, A	Phase Transitions of alpha-FeOOH Above 50 GPa Using X-ray Diffraction	12.2.2	4		
	Lu, T	Elasticity and Strength of Transparent Micro- and Nano-ceramics	12.2.2	21		
Totals				657		
Goal						

Beamline proposals for the July-December 2010 cycle with numbers of shifts requested. The C and GU designations on the top portion of this figure (left hand side) indicate the number of shifts allocated on a preliminary basis to COMPRES users versus General Users. In many instances, since laser-heating was becoming more routine and hence extended user set-up time was no longer necessitated, beamline allocations were substantially reduced below those requested.

B.1.2 COMPTECH "COMPRES Technology Center at Argonne"

Przemyslaw K. Dera[1], Guoyin Shen[2], Quentin Williams[3], Lars Ehm[4], Yanbin Wang[1], Robert T. Downs[5] and Daniel L. Farber[6]

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[5]*University of Arizona at Tucson*
[5]*Lawrence Livermore National Lab*

COMPRES at Argonne and the Concept of COMPTECH

The mission of COMPRES is driven by its science goals, but it is mainly the experimental technology available at central facilities such as synchrotron beamlines, that enables this science. Argonne National Laboratory's synchrotron source, the Advanced Photon Source (APS) is currently the leading central facility for synchrotron science in the United States. With its storage ring operating at 7 GeV electron energy, the experimental stations at APS offer a significantly higher flux of hard x-rays (above 20 keV), which are most suitable for high-pressure experiments, than other synchrotron sources in the country. APS beamlines are heavily utilized by members of the COMPRES community to carry out their research. APS is currently in the initial stage of a major midterm upgrade which will significantly boost the current capabilities and increase the amount of beam time available to users. This will be done by canting most of the existing insertion devices and creating new experimental stations.

Since its inception, COMPRES has maintained its presence at APS by funding Infrastructure Development projects located at high-pressure beamlines (Sectors 3, 13, 16). Many of these projects have been very successful and have had a significant impact on the COMPRES community and their research output, as well as on the APS itself. Thus far COMPRES has not operated any permanent facility at APS, but there are currently interest and plans to pursue Partner User Proposal relationships with several APS beamlines to increase access to these facilities by the COMPRES members, and to enhance novel experimental technology and instrumentation suitable for Earth science at these facilities.

This proposal requests funds to create a COMPRES Technology Center (COMPTECH) at Argonne with the main mission to initiate, facilitate and coordinate new Partner User Proposals to create new capabilities and improve the ease of use of state-of-the art facilities at existing beamlines at APS for the COMPRES community.

The location of the COMPTECH at APS brings several significant advantages: (i) as the most advanced hard X-ray synchrotron source in the country APS hosts a variety on unique, state of the art instruments and engages in development of novel forefront experimental technology (ii) APS hosts several of the current COMPRES projects and has expressed a willingness to continue to partner with COMPRES through PUP proposals; (iii) APS also hosts many resident experts on experimental technology, software and methods development, as well as a broad spectrum of high-pressure researchers, who will be interested in collaborating with the COMPTECH, (iv) the lab is within driving distance from the COMPRES headquarters at Urbana and in close proximity to two major airports; (v) it is located at a central research facility frequently visited by the majority of active COMPRES users, which should stimulate interactions and seed collaborations.

Partner User Proposals

At DOE synchrotron laboratories in the US, the majority of beam time is distributed to interested user researchers through the General User Proposal (GUP) system. Proposals submitted by users are evaluated by subject area Proposal Review Panels (PRP), with regard to scientific importance, expected impact on the field and technical feasibility. Typical GUPs cover experiments that are conducted within one or few visits to the synchrotron and utilize already available experimental technology. In addition to GUPs most synchrotron beamlines also accept Partner User Proposals (PUP). These proposals, in addition to science goals, are usually focused on development of new capabilities or methodologies (in demand by community of users) at existing beamlines, can span over a longer period of time (up to 3 years), require the user to contribute to the project in some substantial way (personnel, instrumentation, or responsibility for some part of the development (e.g. software), and in return, include guaranteed access to some portion (typically up to 15%) of the beam time available at the facility for the duration of the project. PUPs are evaluated not only by the PRPs but also by the APS Scientific Advisory Committee (SAC) and APS management.

PUPs present an excellent opportunity for scientific consortia with broad synchrotron user bases to develop existing state-of-the-art synchrotron facilities in new directions important to their community. The PUP model of developing new beamline capabilities for COMPRES researchers is much more rapid and efficient than funding and operating a new beamline. This will allow COMPRES to have an influence on the direction of the instrumentation and methodology development at the participating beamlines, and at the same time will allow for dynamics in adapting to the most important current needs of the community.

Carrying out several separate PUP projects independently would still be quite costly at the scale of COMPRES' budget, as each project would require a separate independent investment in personnel and/or instrumentation. The main idea behind COMPTECH is to streamline and coordinate these independent projects though creating centralized technology-oriented resources that can be utilized and shared for supporting several PUP projects at once. With the COMPTECH located at Argonne and expected to include in the future several staff scientists, assigning personnel to PUP projects will be much easier than having to fund a new position and find a skilled candidate each time. A significant advantage of having long-term staff, as opposed to typical 2-year term postdoctoral researchers working on PUP projects will be accumulated experience and expertise which will make the contributions of these scientists to new projects much more significant.

Because of the new operational model fpr large scale facilities funded by the US Department of Energy (such as the Spallation Neutron Source (SNS) and the National Synchrotron Light Source-II) and the planned transition of COMPRES operations from the current NSLS to a Partner User Proposal relation at NSLS-II, COMPTECH will serve as model for the creation of a similar center at Brookhaven (and perhaps Oak Ridge at a later time).

The COMPRES Technology Officer position

In order to effectively operate Partner User programs involving several synchrotron beamlines COMPRES will need dedicated and experienced technology-oriented staff. This proposal requests funds to hire an experienced research scientist, above the postdoctoral level to serve as the COMPRES Technology Officer (CTO) and co-PI of the COMPTECH Center at Argonne. This person should have a good publication record, preferably a successful funding history, as well as very strong technical background (synchrotron technology, scientific instrumentation, software development, high-pressure techniques). The CTO will be employed by the University of Illinois at Urbana-Champaign, but will be located permanently at Argonne National Lab (which brings a benefit of a low off-campus indirect cost rate of 24%). The university appointment of the CTO will be at a level that includes grant PI eligibility and allows the CTO to prepare and submit research as well as infrastructure and development grant proposals. The CTO will have four main goals during the five years of this project duration: (i) Establish a successful technical development program (involving instrumentation, method and/or software development) focused on issues relevant to the COMPRES community and emphasizing transferability/portability of the solutions, (ii) Establish a successful research program in mineral physics based on the high-pressure facilities available at the Argonne Lab and (iii) Prepare and submit Partner User Proposals to beamlines at APS that are identified by the COMPRES Executive Committee as important for community progress and are themselves interested in such relationship (currently Sectors 3, 30 and 34 have been identified as potential Partner User Proposal facilities, and have expressed interest in PUP relation with COMPRES) and (iv) Through writing and submitting grant proposals spearhead efforts to raise additional funds needed to carry on effective PUP projects. Additionally, the CTO would serve as a liaison and lobby base for contact and negotiations with the APS administration and management regarding any current or future needs or initiatives that the COMPRES community might identify.

The CTO appointment will be for an initial period of five years, with a possibility of renewal depending on successful performance and availability of funding. The performance of the CTO in pursuing the mission of COMPTECH will be evaluated at the end of year three, and then at the end of the five-year period of this project by the COMPRES Executive Committee, in a manner similar to tenure evaluation at a university. The ability to attract a highly skilled, and very well trained and motivated person for the CTO position is critical for the success of the described COMPTECH enterprise . As a consequence, creating a well-defined long-term career perspective with emphasis placed on both technical, as well as research accomplishments will be very important.

The proposed COMPTECH personnel (1 senior scientist in years 1-5 and a second, more junior scientist in years 4 and 5) and the moderate equipment funds requested (~$100K over five years) will create the necessary seed resources to significantly contribute to 2-3 PUP projects at APS beamlines.

The beamlies which we identified as possible locations of the first three COMPTECH PUP projects (IXS at sectors 3 and 30, nanodiffraction at sector 34, total scattering at sector 1, time-resolved experiments at sector 7) already have, to varying degrees, been involved with research of interest to the mineral physics community or, as in the case of the COMPRES relationship with Sector 3, are part of a successful Earth science high-pressure program. However, all of these facilities have limited staff and struggle with lack of easy-to-use and intuitive instrument control and/or data collection software. These shortcomings are a huge impediment when trying to build a broad community-wide user base for members of mineral physics community. This is in contrast to small groups that currently dominate the usership of these beamlines (largely from condensed matter physics and materials engineering), who focus on specific types of experiments over long periods of time and have developed personalized data analysis tools for their particular research needs. There are many existing software solutions (e.g. single crystal data collection and analysis software developed at GSECARS), which COMPTECH staff could help implement at these facilities, with significant benefit for the experimental capabilities, efficiency of utilization of beam time and scientific output.

The funds requested within this proposal are sufficient to start the three PUP projects outlined in this proposal. It is, however, expected that the CTO will initiate efforts to raise additional funds (either through COMPRES Infrastructure Development proposals or through proposals submitted directly to funding agencies) to supplement and optimize the hardware at the PUP facilities and hire additional personnel for the Center.

Examples of technical development projects for COMPTECH

The exact list of projects and priorities for the technical development activities at COMPTECH will be set by the Technology Advisory Board with input from the Executive Committee. Community input will be obtained through regular solicitations for feedback and suggestions, and via regular town-hall sessions at the COMPRES annual meeting (and prospectively at other meetings, such as AGU). The focus of the developments will be to create solutions that are most needed by the community at the moment, will be used by more than one facility, and which will be easily transferable. Because of the evolving needs of the community, we anticipate a similar evolution in the specific development projects that will be pursued: we envision a broad suite of potential projects, ranging from combining advances achieved at different facilities (for example, coupling improvements in pressure/temperature technology with improved spectroscopic or diffraction measurements), to making any of a range of novel x-ray spectroscopic or diffraction measurements more generally accessible to geosciences users. The number of active projects at any given time will depend on the number of COMPTECH personnel (additional personnel expected to be funded from external grants to be headed by the CTO). A few examples of projects worthy of immediate attention include:

Studying phonons with non-resonant momentum-resolved inelastic X-ray scattering

The study of phonon dispersions in condensed matter characterizes collective atomic motions and provides insight into many physical properties, such as elasticity, as well as interatomic forces, structural phase stability and transitions, anharmonicity, and electron correlation. In situ experiments that constrain the elastic tensor and acoustic wave velocities in minerals at high pressure and temperature conditions are extremely important for geophysics as they provide a direct link between microscale phenomena, studied in the lab, and macroscopic seismic observations, which remain our major window into deep-earth composition, structure and properties.

There exist a range of conventional techniques that are traditionally used for *in situ* studies of acoustic phonons, including Brillouin spectroscopy and ultrasonic interferometry. COMPRES has made significant investments in these capabilities by funding the online Brillouin spectrometer at GSECARS and the LVP interferometry program at NSLS. While these conventional techniques remain invaluable, and have stimulated numerous high-profile discoveries in mineral physics, they also have significant limitations in terms of sample preparation, surface quality, attainable pressure range as well as the ability to precisely characterize samples of low symmetry.

In the last decade, new synchrotron-based *in situ* diamond-anvil cell techniques based on momentum-resolved nonresonant inelastic X-ray scattering (IXS) have been introduced that allow the study of acoustic phonons. The IXS experiments require 3-rd generation synchrotrons and high energy resolution (1 meV range). Classically, IXS experiments are performed on high-quality single crystals where all the independent vibrational branches can be accessed. Single-crystal IXS allows all of the independent elements of the elastic tensor to be directly determined from the initial slope of the phonon dispersion of selected longitudinal acoustic and transverse acoustic modes, without any external input or *a priori* mode. IXS overcomes the limitations of previously used surface-sensitive techniques that require a complex data inversion (e.g., surface Brillouin), involving modeling of the sound waves at the interfaces and input of external parameters to obtain bulk properties. Single-crystal IXS offers several significant advantages over Brillouin scattering in the diamond anvil cell: (i) it does not require the sample to be transparent; (ii) the quality of data does not depend on the sample surface perfection; and (iii) data collection does not require knowledge of the refractive index, nor sample surfaces that are parallel to the diamond anvil surfaces (allowing the exploration of more independent q-wave vectors of the sample).

For the Earth Sciences, single-crystal experiments have two drawbacks, 1) the requirement of high-quality single crystals, which for many high pressure phases are not available, and 2) the technique is very time intensive, requiring weeks of beamtime to measure a reasonable set of phonon branches at even a few pressures.

Recent developments in IXS techniques have produced a number of approaches that could be exploited to provide critical data from polycrystalline samples either as an end in itself or as a precursor to more detailed point by point measurements (we note that thermal diffuse scattering can also be utilized to extract essentially multiplexed data; however, this technique also requires single crystals and there are other efforts pursuing this direction for high pressure lattice dynamics). Of these we see two important support programs for both single crystal measurements as well as polycrystalline samples techniques and two specialized programs for the extraction of data from polycrystalline samples.

1) A basic set of symmetry-based crystallographic software for the prediction of phonon intensities and scanning directions given a sample orientation matrix.
2) Access to and integration of basic *ab initio* tools into the experimental development program to predict the position and behavior of phonon modes as a function of intensive parameters [1].
3) Standardized software for the extraction of the phonon density of states from powder samples [2-5].
4) Software for the extraction of full phonon dispersions from measurements on polycrystalline samples [1, 6].

Clearly, it is too large a burden and not the most scientifically expedient path forward to expect every user to develop these capabilities on their own. The software listed is extremely complex. Indeed, the best approach would be to have available a set of tools that would open these experimental directions to the broader scientific community. Thus, we argue that relatively small investments in human capital to develop the scientific software would facilitate access to these new techniques in a cost effective way. This would enhance the investments that the NSF and DOE have made in the IXS infrastructure at the APS. Further, while the broader IXS community has long recognized this general need for integrated analysis tools, none of the other IXS facilities around the world have been able to invest in, nor capitalize on the recent theoretical developments. Thus, an effort towards building computational infrastructure around our existing IXS hardware is both timely and would leapfrog the APS (and, by association, COMPRES and high-pressure Earth Sciences) into a leadership position in the analysis of IXS data. Such a set of tools would not only facilitate a new class of experiments but would have an especially large impact on the types of properties we could extract from earth materials at extreme conditions.

We propose to pursue the above developments within the scope of collaboration of the COMPRES Technology Center with Sectors 3 and 30 at APS.

Development of data analysis software for in-situ and ex-situ characterization of crystal structure, chemical composition and morphology and strain/stress state of sub-micrometer crystallites

Advances in x-ray optics and synchrotron technology make possible focusing of the synchrotron beam to smaller and smaller sizes. Currently, leading nanodiffraction beamlines, including Sector 34 at APS, offer nanofocusing capabilities reaching 200 nanometers. This progress opens very new and exciting opportunities in high-pressure science making possible experiments at much higher pressure conditions (above 2 megabars) and minimizing gradients (both pressure and temperature, e.g. during laser heating experiments). Even with samples composed of multiple phases or characterized by chemical heterogeneities, nanobeam techniques offer possibilities to obtain information about the structure and composition of the individual grains without the complications of overlapping signals.

At length-scales below 1 micrometer the standard definitions of single crystal and powder are no longer relevant, as most grains in polycrystalline material are comparable with, or even larger than the nanobeam size. As a result, the statistical requirement for good quality powder data, which demands the powder to contain as many as 10^6 grains, can never be satisfied. Conventional single-crystal methods utilizing sample rotation and a monochromatic beam cannot be easily combined with nanobeams either, as no available mechanical solutions allow sample rotation with a sphere of confusion below 1 micrometer.

In response to this challenge, the past decade witnessed significant efforts in development of methods known as 3D X-ray microscopy. Several alternative approaches have been developed at Sector 34, APS, ORNL, Risø National Laboratory (Denmark) and ESRF (France), with a focus mainly on materials science and chemistry applications. Among these approaches, a combination of Laue diffraction and energy scanning seems the most compatible and promising solution for high-pressure science. The first successful tests and pioneering experiments utilizing Laue microdiffraction have been conducted both in the diamond anvil cell and the large volume press (in the latter case the same approach is used, but with much larger beam and sample sizes). While the progress in this field has been very impressive and exciting, and many high-pressure scientists are becoming increasingly interested in implementing nanobeam experiments in their research (Sector 34 has an on-going high-pressure user program involving CIW and UNLV), the data analysis solutions available today do not yet offer full functionality for identification and structure determination of yet unknown phases (e.g. produced as a result of a phase transition or high-temperature chemical reaction or incongruent melting) [7-8].

We plan to establish a formal collaboration between COMPRES researchers and APS Sector 34 in the form of a Partner User Proposal (PUP). The aim of the PUP will be to bring the most exciting high-profile mineral physics experiments to the nanodiffraction facility and contribute to further development of the experimental methodology (focusing on the high-pressure specific solutions) as well as data analysis methods including ab-initio structure determinations of unknown phases from submicrometer sized grains and strain/stress mapping in polycrystalline aggregates. The latter application was identified as one of the main new technologies of interest to the proposed 4DE high-pressure beamline at the NSLS-II. The role of the CTO in this collaboration will be to seek funding for a full-time postdoctoral scientist to be assigned to Sector 34 and to contribute to the development of data analysis tools for the tasks described above.

Developments of synchrotron based in situ capabilities to study atomic structure and properties of highly disordered and non-crystalline Earth materials

Advances in high-pressure technique and synchrotron instrumentation have led to the development of robust structure-property relationships in crystalline Earth materials. However, knowledge of the structure and properties of highly disordered and non-crystalline Earth material at pressure and temperature conditions of Earth's interior are is comparatively primitive. The lack of experimental data on these important classes of Earth materials limits our understanding of important topics in Earth structure and evolution such as the formation and ascent of magma, the origin of large igneous provinces, the role of magmatic liquids in mass and heat transfer in Earth's interior, and the influence of disorder on the elasticity of minerals.

Recent developments in high-energy synchrotron instrumentation in conjunction with the methodological improvements in X-ray total scattering now allow the study of atomic arrangements in highly disordered and non-crystalline Earth materials [9-11]. In conjunction with current efforts at GSECARS and HPCAT on *in situ* property measurements in melts, an opportunity to develop robust structure-property relationships for disordered and non-crystalline Earth materials is emerging. However, some challenges on the experimental and data evaluation side remain, such as: (i) reliable inert containment of liquids in pressure vessels, (ii) improvement of angular access in pressure cells, and (iii) development and/or adaptation of data evaluation methods based on large atom assemblies (e.g. Reverse Monte-Carlo approaches).

The role of the CTO in this project will be to (i) engage in collaborations with researchers at APS Sectors 1 (High-Energy X-ray Scattering beamline) and Sector 11 (Dedicated Pair Distribution Function and High-energy Diffraction beamlines) in the form of a Partner User Proposal to further develop in situ X-ray total scattering technique, (ii) link to the existing programs on property measurements of non-crystalline Earth materials at GSECARS and HPCAT, and (iii) engage and coordinate with the program at X17B3 at the National Synchrotron Light Source (NSLS) on optimization of pressure cells, data evaluation and data based modeling. This project will bundle efforts that are currently progressing independently at APS and COMPRES supported beamline X17B3at NSLS. Furthermore, the instrumentation and methodology development will be beneficial for the scientific program at the proposed high-pressure beamline 4DE at NSLS-II. We expect that the CTO in collaboration with the leader of the X-ray total scattering effort at NSLS will seek funding for a full-time postdoctoral scientist to be based at APS and to participate in the above described effort and the coordination between APS and NSLS scientists.

Management plan

The management of the COMPTECH project will be carried out in a way similar to all the other COMPRES facilities. The budget of COMPTECH will be managed through an account, at the University of Illinois (separate from the main COMPRES accounts), where PI Dera holds an adjunct Associate Professor position.

5.1. Technology Advisory Board (TAB)

Since the mission of COMPTECH is to represent a broad community of mineral physics researchers, COMPRES will appoint a Technology Advisory Board (TAB) to oversee and guide the activities of COMPTECH. TAB will be comprised of synchrotron technology experts, members of the central high-pressure facilities (e.g. HPCAT, GSECARS), managers of the other COMPRES facilities

(ALS, NSLS) and mineral physics researchers representing a cross-section of the COMPRES community. The initial membership of TAB will be comprised of a chair plus six members (the co-PIs of this proposal), with the PI of this proposal serving as the initial TAB chair. President of COMPRES as well as chairs of the two standing committees will serve as *ex officio* members of the TAB.

The TAB will oversee and coordinate all other COMPRES efforts at non-COMPRES beamlines, such as the IXS and Mossbauer project at Sector 3, and the COMPTECH efforts at the NSLS-II.

The term of membership in the TAB will be limited to five years for the chair and three years for the members. Both chair as well as members can be re-elected for one additional term. Candidates for new TAB member appointments will be submitted by the TAB chair and CTO for approval by the COMPRES Executive Committee.

TAB will meet once per month through teleconferencing to discuss the activities and progress of the project as well as to plan future initiatives and actions.

The chair of the TAB and the CTO will serve as co-PIs of the COMPTECH project and will be co-responsible for preparing COMPTECH annual reports.

In years 4 and 5 of COMPRES-III an extension of the Technology Center is planned with a CTO position at Brookhaven National Laboratory, similar to the COMPTECH at Argonne. The Brookhaven CTO will be run through Stony Brook (Weidner, PI). The Technology Advisory Board of COMPTECH will also serve as the managing body for the BNL Technology Office. Having a common advisory board will coordinate efforts between the two labs in a way that takes advantage of the unique capabilities of each facility, avoids unnecessary duplication of efforts, and puts personnel effort where it is most needed.

5.2. Reporting

CTO and the chair of the TAB will prepare annual reports to be reviewed by the COMPRES Facilities Committee and approved by the Executive Committee.

Outreach and education

In the process of developing new, unconventional techniques with novel experimental capabilities, an effort to build a wide community of users requires outreach and education. The community must be informed about new capabilities, trained in general aspects of experiments. Making people aware of exciting new tools for augmenting their research agendas is key for maximizing the impact of a developmental effort on the quality of science. The mere existence of an advanced tool that nobody knows how to use is a waste of resources. COMPRES and the co-PI of this proposal have significant experience and a successful track record in educating and informing the community about new experimental techniques, and in broadening the use of synchrotron-based research. COMPRES frequently sponsors workshops related to its Infrastructure Development Projects. Dera and Wang organized and chaired such topical workshops on Single Crystal X-ray Diffraction at Megabar Pressure (Chicago, 2005) and Large Volume Press Techniques (Chicago, 2007). The 2005 workshop resulted in publication of special volume of Journal of Synchrotron Radiation on “High-pressure Crystallography”, and the 2007 workshop resulted in a special volume in the journal High Pressure Research on “Synchrotron High-Pressure Mineral Physics and Materials Science”. Dera also served as a director of 2009 International School on High-pressure crystallography in Erice, Italy (proceedings published as a volume in NATO Science for Peace and Technology Series B in 2010). Shen organized CDAC (Carnegie-DOE Alliance) summer schools for undergraduate and graduate students in 2009 and 2010. Downs co-edited a volume in Reviews in Mineralogy and Geochemistry on High-pressure and high-temperature comparative crystal chemistry. From 2008 to 2010 Ehm organized three workshops at Brookhaven on the planning of new beamlines for mineral physics at NSLS-II. Ehm also co-organized workshops at Brookhaven National Laboratory on” Nanoscale Diffraction of Materials” (May 2010), “Supercritical Carbon Dioxide Materials Interactions” (March 2011), and “X-ray Diffraction and Spectroscopy to Study Dynamic Phenomena under Extremes” (May 2011). Ehm is a Co-PI on a recently funded Track 1 proposal of NSF’s

Opportunities for Enhancing Diversity in the Geosciences program titled "A career path for African-American Students from HBCUs to National Laboratories."

With this combined experience in E&O we plan similar efforts to attract a large community of new Earth science users to the facilities that are developed by COMPTECH. In particular, COMPTECH will organize regular workshops at the COMPRES annual meetings to educate and train and future users of the PUP facilities. For each PUP project we will organize a separate dedicated workshop at Argonne (with the likelihood of published proceedings with international circulation). The CTO will regularly present the progress reports of each project as posters at the annual AGU meetings. The software developed at COMPTECH as well as information about new experimental capabilities at participating beamlines will be distributed through a COMPTECH website.

Through its association with Argonne National Lab COMPTECH will participate in the Argonne-wide summer internship for undergraduates program (funded by DOE). Members of the TAB have extensive experience with supervising both Argonne Interns as well as participating in REU programs at their home institutions.

Relation of the COMPTECH with GSECARS, HPCAT, HPSynC

There are currently several organizations at APS that focus on and promote high-pressure science, and serve the COMPRES community (among other supported communities). These include GSECARS, HPCAT. HPSYNC is focused on high-pressure but with a primary emphasis on materials sciences. When considering the COMPTECH concept, it is important to recognize and define the compatibilities and differences with all of the above to evaluate the benefits of funding COMPTECH, We do not want to duplicate the functionality of an existing and enterprise.

The mission and scope of HPCAT are focused on multidisciplinary high-pressure science, with a significant, but not dominant, geoscience component. HPCAT operates its own facility (Sector 16 at APS) that features four experimental stations offering a range of high-pressure experimental capabilities. Members of the COMPRES community access HPCAT via the 25% of total beamtime that is distributed through General User Proposals (GUP) on a competitive basis. 75% of HPCAT beamtime is distributed to HPCAT members.

GSECARS is a geoscience-focused organization, with a program that, in addition to high-pressure mineral physics, encompasses aspects of environmental chemistry, mineralogy, petrology, and aqueous geochemistry. Mineral physics constitutes approximately 50% of the GSECARS program. 100% of total beam time in 4 experimental stations is available to general users on a competitive basis. The emphasis of COMPTECH activities will be on experimental techniques not available at GSECARS.

Both HPCAT and GSECARS have active and successful development programs in experimental methodology, instrumentation and software development,. Most of these focus on developing in-house state-of-the-art solutions to be utilized at their own beamlines, and are driven by beamline specific interests and priorities. There are a few very important examples of the development of shared/transferable technologies, including EPICS and IDL software by M. Rivers, X-ray focusing optics by P. Eng, laser-heating and temperature calibration software by G. Shen, and the COMPRES-sponsored Gas loading Apparatus. However, GSECARS and HPCAT scientists are funded to design develop and maintain experimental facilities within their own organization. Neither GSECARS nor HPCAT engage in Partner User relations with other beamlines at APS. Because of the user support and facility maintenance duties, as well as their own research programs, the scientists at GSECARS and HPCAT are able to devote only a small fraction of their time to shared technology development.

The HPSynC organization is not attached to a particular facility. It is with a multidisciplinary science program focused on high-pressure physics and materials science with an emphasis on energy related issues. HPSynC devotes part of its efforts to development of portable/transferable solutions for high-pressure synchrotron experiments and collaborative research. Geoscience is a rather minor component of HPSynC activities, and HPSynC does not engage in Partner User relations with APS beamlines.

The COMPRES Technology Center at Argonne will differ from and be complementary to the three above described organizations in the following ways:

- 100% focused on geoscience and mineral physics.
- Actively engaged in collaborations with new synchrotron beamline facilities through Partner User Projects..
- Priorities for research and development are set by the COMPRES community through the Technology Advisory Board.
- Free from regular user support and facility maintenance duties, COMPTECH personnel will be able to devote a more significant portion of their time to developments identified as COMPRES priorities.
- Significant effort (50%) devoted to development of transferable, shared instrumentation, methodology and software solutions for high-pressure geosciences. Much of this will be done in collaboration with existing experts at GSECARS, HPCAT and HPSynC.
- Outreach and Education will be a significant part of the COMPTECH mission. COMPTECH will be a community resource. Developed infrastructure will remain at beamlines through PUP agreements, for the continued benefit of all. Software developed by COMPTECH will be openly and freely available. Resources will be made available to help new users develop competitive proposals for beamtime and to help with make their initial experiments successful. Effort will be put into developing a broad geoscience high-pressure user base through workshops and online resources.

We feel confident that the COMPTECH concept will be effective at enabling new synchrotron capabilities for high-pressure Earth sciences applications and building a broad user base for those capabilities. This approach builds upon the experience gained with the highly successful COMPRES Infrastructure Development project at Sector 3 of the APS, where COMPRES greatly accelerated the use of Nuclear Resonant Inelastic X-ray Scattering and Synchrotron Mossbauer Scattering within the entire Earth sciences community. The Earth science user base for Sector 3 is now broad, which has led to much new science from many distinct user groups. These efforts can now be extended on a larger scale to a number of beamlines and techniques at the APS and later at the NSLS-II.

In order to effectively coordinate the activities of all the high-pressure related organizations named above, the initial membership of the TAB includes representatives of GSECARS (Wang and Dera), and HPCAT (Shen).

We anticipate that the COMPTECH will receive the lab/office space from APS (letter of support from APS is attached as a supplementary document).

The requested investment on the side of COMPRES to create the COMPRES Technology Center at Argonne is a little larger than a typical Infrastructure Development project, however it is expected to be a very high-return investment as it will create (i) a technology center including several personnel funded from sources other than the COMPRES budget (ii) will create a route (a full-time salaried and experienced CTO with PI eligibility) for COMPRES to approach funding agencies other than NSF-EAR IF (which funds COMPRES) for important new community projects (iii) will create a means to better coordinate the research and development efforts at various COMPRES facilities (also outside APS) and emphasize development of transferrable technologies that create significant value added.

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B.1.3 HIGH PRESSURE PROGRAM AT THE NATIONAL SYNCHROTRON LIGHT SOURCES

The high-pressure x-ray program at the national Synchrotron Light source is currently located at superconducting wiggler beamline X17 and consists of three experimental endstations and a support laboratory. The endstations X17B3 and X17C provide experimental capabilities for diffraction experiments using diamond anvil cells (DAC) as pressure generating devices. X17B2 is equipped with a 1000 ton press and several inserts for different sample geometries. Furthermore, a monochromatic side-station has been established at X17B2 for high-pressure experiments in a Paris-Edinburgh compact large volume pressure cell.

The high-pressure program at X17 is the longest continuous operating high-pressure program at United States synchrotron radiation facilities. Over the years of operation it has been a center of innovation for the novel experimental techniques and development of high-pressure equipment. For example: (i) X17B2 has been the first endstation in the US with a multi-anvil high-pressure system (ii) the first *in-situ* laser heating experiments at high-pressure have been conducted at X17C, (iii) the first simultaneous high-pressure and temperature experiments at mbar pressures have been conducted at X17C. This culture of scientific and technological innovation remains active at the program as the recent accomplishments and resulting scientific highlights attest.

COMPRES is a Contributing User (CU) at the X17 facility beamline at the NSLS. The NSLS is responsible for the operation of the beamline (optics, safety systems, etc.) while COMPRES is responsible for operation of the experimental stations. 50% of the beamtime is given to general users (GU) and 50% of the available beamtime is assigned to COMPRES. All proposals are first submitted through the competitive proposal system at NSLS for GU time. CU time may be assigned to COMPRES proposals that were not assigned GU time, to increase the number of days for a successful GU proposal, or for use by beamline staff.

At this time, a new synchrotron is being built at Brookhaven National Laboratories, just across the street from the NSLS. The NSLS II synchrotron, Figure 1, is designed to set new standards in brightness of beam yielding improvements in spatial resolution (1 nm) and energy discrimination (0.1meV). Construction has passed the half-way point. The time line is given in Figure 2. We anticipate that the NSLS will close during 2014 and that the NSLS II will open to general users in 2015. These dates fall in the time frame covered by this proposal. In this section we first discuss the continued operations at the NSLS. We present the multi-anvil and the diamond anvil systems separately. Then we present the current vision of COMPRES at the NSLS II. Diamond cell and multi-anvil cell efforts are merged into one high-pressure section. The NSLS phase is an important one as we can take advantage of a mature experimental set-up and at the same time pursue important preliminary studies that will take advantage of the NSLS II. The NSLS II phase is extremely exciting as new capabilities lay ahead of us and we have a unique opportunity to integrate high pressure into the entire synchrotron ring. We already have commitments for deploying multi-anvil and diamond anvil high -pressure systems on a beamline that is scheduled to be available on day one.

In this five-year time covered by this proposal, we will continue to operate the X17 high-pressure program at the NSLS in the endstations X17B2 for large-volume high-pressure research and at X17B3 and X17C for the diamond anvil cell research for the next two years (2012-2014) for the COMPRES community. Donald Weidner will serve as PI of the multi-anvil program and Tom Duffy and Donald Weidner for the diamond anvil cell program for the next two years. Additionally, we propose the creation of a COMPRES Technology Center at Brookhaven National Laboratory (CTCB) with one COMPRES Technology Officer (CTO), who will be connected to the X17 high-pressure program, but not involved in the day-to-day operation. The CTO will have three main goals: (i) Establish a successful technical developments program (involving method and instrumentation development) focused on issues relevant to the COMPRES community and transferable to NSLS-II and/or other US synchrotron radiation facilities, (ii) initiate collaborations with the lead scientists of NSLS-II beamlines currently under design/construction, which may have a relevance for the COMPRES community and explore solutions to integrate high-pressure Earth Sciences research into the beamlines research/user program, and (iii) establish a successful mineral physics centered research program that takes advantage of the existing capabilities at NSLS and upcoming facilities at NSLS-II. The CTO will spearhead the effort to transfer high-pressure technology to suitable beamlines around the NSLS II ring.

The high-pressure program at X17 will transfer in October 2014 to the D-hutch of the X-ray Powder Diffraction Beamline (XPD) at NSLS-II. Donald Weidner and Lars Ehm will serve as co-PI's for the joint multi-anvil and diamond-anvil program for the following three year period from 2014-2017.

Beamline development workshops

The High Pressure community has held four workshops at Brookhaven National Laboratory to refine and discuss future developments of high pressure at the NLSL in anticipation of NSLS-II:

- February 25-26. 2006. COMPRES and MPI (Stony Brook University) sponsored "NSLS X-Ray High Pressure Research Workshop: Current operation and vision into NSLS II"
- July 17, 2007, there was a High Pressure discussion as part of an NSLS-II workshop
- January 17-18, 2008, there was a breakout session during the joint workshop for "The NSLS-II Powder Diffraction Project Beamline" and "Materials Science Engineering Strategic Planning for NSLS and NSLS-II"
- April 29-30, 2010 workshop for preparation of proposal for "4D Studies in extreme environments (4DE2)", sponsored by COMPRES and Brookhaven National Lab
- March 21-24, 2011, Materials Interaction with supercritical CO2, sponsored by Mineral Physics Institute, Stony Brook and Brookhaven National Labs.
- The program of this proposal is an outgrowth of these workshops.

Figure 1 The NSLS II constructions site March, 2011

DOE Project Milestone Schedule

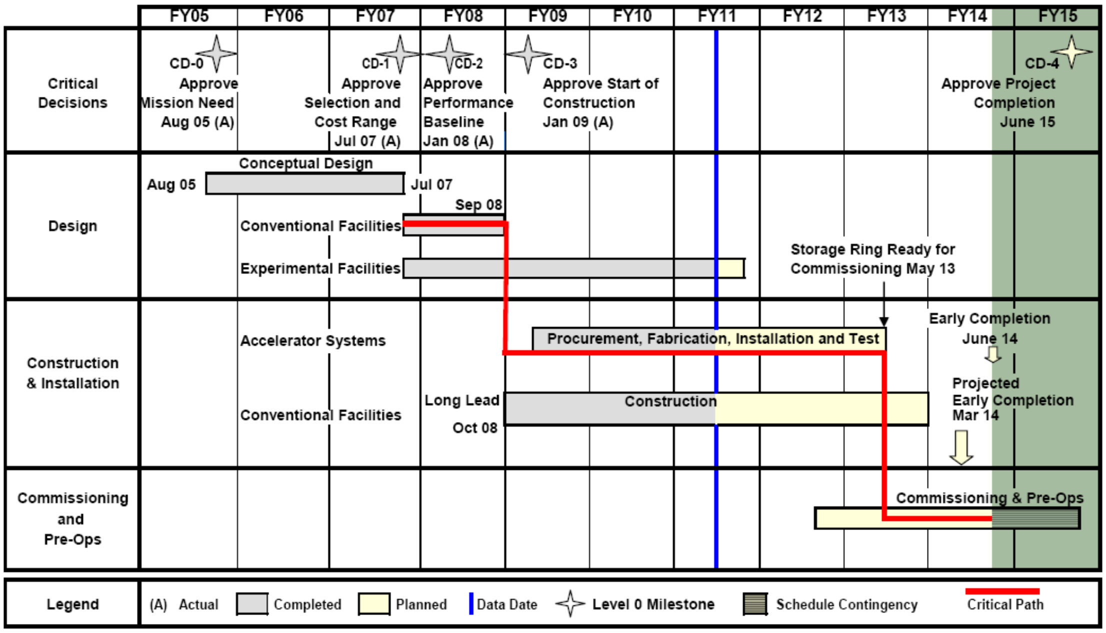


Figure 2 Time line for the construction of the NSLS II. Target for user operations is October, 2014.

B.1.4 Multi-anvil High Pressure Facilities at National Synchrotron Light Source

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 studies. Here we focus on the large-volume system at the NSLS. 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 is the first beamline in the world to measure Q of elastic deformation at seismic frequencies at pressures of several GPa. This beamline continues to contribute to high-pressure crystallography, phase equilibrium, and equations of state.

Scientific Program

Our understanding of the makeup and evolution of the Earth is strongly tied to our understanding of the materials that make up the Earth. Pressure and temperature set the environment where these minerals and melts are found, and it is the goal of high-pressure experimental studies to probe and characterize Earth material systems. A 2008 workshop on seismological research frontiers *(Seismological Grand Challenges in Understanding Earth's Dynamic Systems, 2009)*, funded by the National Science Foundation (NSF), considered promising research directions for the next decades and identified 10 Seismological Grand Challenge research questions including: *How do faults slip?; How does the near-surface environment affect natural hazards and resources?; What is the relationship between stress and strain in the lithosphere?; Where are water and hydrocarbons hidden beneath the surface?; How do magmas ascend and erupt?; What is the lithosphere-asthenosphere boundary?; How do plate boundary systems evolve?; How do temperature and composition variations control mantle and core convection?;* and *How are Earth's internal boundaries affected by dynamics?* While these questions drive the agenda of the next decade seismology research, they are also central to the research program of the multi-anvil high-pressure beamline at NSLS.

Elasticity

The most robust fingerprint of the chemical and thermal state of the Earth's interior are the elastic properties of 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 database 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. The multi-anvil beamline at the NSLS has been the pioneer of such measurements and continues to develop a wider array of possible samples for such high P-T experiments[3, 6, 9, 10, 30-34, 48, 49, 55-59, 74-76, 78-82, 84-89, 91, 123-126]. An example is the recent study of $MgSiO_3$ perovskite to 9 GPa and 873 K [58]. In principle this technique can be extended to 25 GPa and 2000K and we plan to reach this capability in the next 5 years.

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. 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. 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. For instance, the lateral and radial variation of viscosity have an important influence on the convection pattern and generation of deep earthquakes, whereas the solubility and diffusivity of elements in various phases control the chemical evolution associated with mantle convection. 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. 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.

The rheology experiments associated with the NSLS beamline have set an entirely new range of conditions for these measurements. We can now conduct uniaxial stress deformation experiments at 10 GPa and 2000K with near the precision of experiments at 0.3 GPa a decade ago. We soon should have the capability to extend these measurements up to 25 GPa with the equipment now being installed at the

NSLS. Through these developments, not only can we infer pressure dependence of mineral properties, but we can examine the properties of high-pressure mineral phases that were impossible before. The technical developments have enabled studies on the relevant properties of minerals [1, 2, 8, 18, 24, 25, 29, 36-47, 60-63, 67, 68, 70, 92-96, 101-105, 118, 120]

The new pressure cells and measurement tools also allow a wide array of new characterizations that are still being explored including measurements of phase transition kinetics through stress oscillations with frequencies in the seismic zone [62, 63, 121], Q measurements at high P and T [65, 66, 69], thermal diffusivity at high P and T [21-23]. New science will emerge as these tools are used to study polycrystalline samples, partially molten samples, and single crystal samples. This next five years promises to be a time of great discoveries, taking the tools we have on hand and pushing our understanding of relevant materials.

Technical advances

The last five years has seen an ever-increasing capability of the experimental program to address significant issues in the Earth sciences. This has come about largely because the user program has included several research groups that have worked with the beamline team to develop many of the new technologies. Some of the advances have come through funding outside of COMPRES such as the 'Grand Challenge' program. Here we summarize some of these advances.

Rheology experimental technique breakthrough

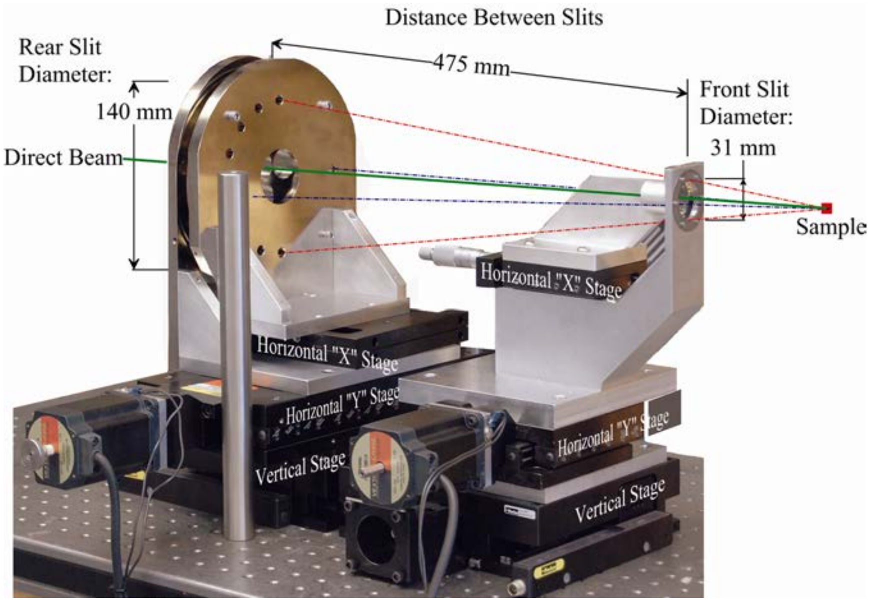
Experimental methods to quantitatively characterize the stress – strain relation in materials at conditions beyond the elastic regime have been limited to pressures of a few hundred MPa. This limits our understanding of the deep Earth not only because we can't simulate the effect of pressure on these properties, but also because we can't study the relevant phases of the deep Earth. In this regard, even pyrope garnet is beyond the reach of these investigations. Over the last decade, the NSLS high-pressure program has pioneered the tools that have moved the realm for such studies from the 100's MPa to 10's GPa. The necessary tools for these studies include 1) a compression system that has the capability of adding a well defined deviatoric stress onto the sample, 2) a means for accurately measuring the magnitude of the deviatoric stress, and 3) a means of measuring the strain in the sample. All of these need to be accomplished with the sample at high pressure and temperature and monitored as a function of time.

D-DIA

A new high pressure-deformation apparatus, called the Deformation DIA (D-DIA), has been married to the synchrotron x-ray source. This new apparatus made its first appearance on a synchrotron beamline at X17 of the NSLS in 2002. In the last five years, its use has matured and it has achieved success beyond the initial hope. Not only has it hosted deformation experiments through its capability of applying a constant uniaxial load to the sample, it has been capable of providing a sinusoidal stress field that has enabled studies of kinetics of phase transition and measurement of Q.

Rotational Drickamer Apparatus

Large shear strains can be applied to a torus shaped sample by rotating the top relative to the bottom. Karato's group has designed and employed a high-pressure system that takes advantage of this process. The Rotational Drickamer Apparatus (RDA) has been developed and used at X17 to study the rheological properties of many mantle materials under high-pressure and temperature conditions. The experimental studies involve three components: (1) development of a new type of deformation apparatus, (2) applications of x-ray facilities at X-17B at the NSLS, and (3) the development of a theoretical model to interpret x-ray diffraction data. In 2004, they started this series of experiments in collaboration with the group at Stony Brook University who has developed all the x-ray facilities needed for this work. The new theory is used to interpret x-ray diffraction data in terms of stress level and its distribution. The maximum pressure and temperature range explored so far is P up to ~22 GPa and T up to ~2200 K. Using this technique, we have determined (1) the pressure dependence of creep strength of water-free olivine to P~10 GPa and T~1900 K, (2) obtained data on the creep strength of wadsleyite and ringwoodite (to P~22 GPa, T~2200 K). These results help to understand the stability of deep continental roots, and the energy dissipation associated with deep slab deformation.



Detector

In situ measurement of stress in polycrystalline samples forms the basis for studies of the mechanical properties of materials with very broad Earth science and materials science applications. Synchrotron x rays have been used to define the local elastic strain in these samples, which in turn define stress. We have developed a new, energy dispersive detection system for white radiation, which has been installed at the National Synchrotron Light Source [117]. The new system provides differential strain measurements with a precision of 3×10-5 for volumes that are 50×50×500 microns. This gives a stress precision of about 10 MPa for silicate minerals. This system enables accurate steady-state, high pressure and high temperature rheological studies of Earth minerals at mantle conditions.

Sinusoidal stress fields

A new data collection protocol for forced oscillation experiments using a multi-anvil high-pressure device can now be carried out with the D-DIA. We derive stress of the sample at high pressure and temperature from synchrotron X-ray diffraction that is synchronized with sample strain measurements from X-ray radiographs. This method yields stress directly from the sample rather than a stress proxy. Furthermore, the diffraction pattern yields useful information concerning time evolution of structurally related phenomena.

The time evolution of a system in a sinusoidal stress field potentially yields a rich array of information ranging from kinetics, anelasticity, lattice preferred orientation induced by grain rotation. X-ray diffraction data are useful for extracting this information at high pressure and temperature. In high-pressure dynamic loading experiments [69, 117], stresses are often inferred from the strain of a stress-proxy such as alumina. In this protocol [69], the strain is determined from length changes of the sample and proxy, which are recorded in X-ray images. The data acquisition takes about a millisecond per image. The attenuation can be resolved for periods as short as 10 seconds.

A more assumption free measure of stress comes from X-ray diffraction (XRD) of the sample [118], with the acquisition time close to 500 seconds. However, the beam condition for XRD requires smaller spot size (50μm x 50μm) than X-ray radiograph (2mm x 2mm), and therefore cannot be done simultaneously.

We have developed a new strategy of time-synchronization among strain, diffraction- based stress, and applied force-field which enables the experiments when the period is longer than 500 seconds [65]. By using X-ray diffraction and imaging, we are able to examine time resolved elastic strain and total strain, we can compare differential stress derived from the sample with that from a proxy, we can examine changes in lattice preferred orientation in-situ in an oscillating stress field at high pressure.

Side station

Ever increasing pressures for more beamtime have motivated us to build the capability to run two experiments simultaneously in the same hutch. The second experiment is made possible by using a single-bounce monochromator that deviates a monochromatic X-ray beam at a slight angle from the main beam. This deviation allows the two beams to separate and provides space to insert a second station beside the main station. We have designed and built a differential stress system based on the T-cup design. We engaged the manufacturers of the Paris-Edinburgh cell to build the press and guideblock. We use an in-hutch imaging plate area detector to record the diffraction pattern. This system is now available for experiments and should be able to host a similar type of experiment as the D-DIA.

Ultrasonic measurements at high pressure and temperatures

At X17B2 of NSLS we have developed techniques to conduct simultaneous ultrasonic interferometry, X-ray diffraction, and X-radiography imaging measurement on solids and liquids at high pressures to 20 GPa and high temperature to 1773K. A series of experiments on minerals relevant to the Earth's deep interior as well as other functional materials under high-pressure and temperature have been performed, and unprecedented data have been obtained. It is worth noting that similar techniques implemented at other synchrotron facilities around the world have all used the X17B2/NSLS facility as a model experimental set-up. The greatest advantage of the current setup is that a combined analysis of the ultrasonic velocity and X-ray diffraction data on crystalline materials provides a unique means to determine the thermoelasticity with a direct determination of pressure using the sample itself. These techniques can also be applied to (1) reliably determine the density equation of state for glass/amorphous materials through the measurements of ultrasonic velocities with direct sample length measurements (e.g., see data on ZrW_2O_8 [90], (2) establish absolute pressure scales, and (3) conduct in-situ investigation on time-dependent processes such as phase transformations and melting. The newest development has enabled us to conduct these measurements up to 20 GPa in pressure and 1773 K in temperature.

Thermal diffusivity at high pressure and temperature

A new method was derived to measure thermal diffusivity of samples at elevated pressure and temperature [21-23]. X-radiograph images of a sample are analyzed to define the distance between two wires as a function of a time while varying temperature. The phase lag of the line separation is determined as a function of radius. This phenomenon represents the thermal pulse moving into the sample from the furnace. Thermal diffusivity is determined from the motion of the pulse. With this technique, thermal diffusivity is measured for many materials relevant to the Earth's interior.

Technical advances for the next five years

1. ***1000 Ton press***: Up till now, the everyday high-pressure system at the multi-anvil beamline has been a 200 ton press with the D-DIA installed in it. We are now changing this to a 1000 ton press with an opening that will allow interchange of high-pressure tooling. The 1000 ton press will allow higher pressure and can work with a Kawai type of 2-stage high-pressure system. This system, complete with motorized positioning table, has been constructed this year and be available for operations in the summer of 2011.
2. ***DT25 guideblock***: In the new press we will soon install a modified Kawai system that will have the capability of providing a differential load to the sample while it is at high pressure and temperature. The anvil cube is 25 mm on this system and so will be very similar to the standard laboratory Kawai pressure system. The main difference is that the upper and lower anvils are pushed by an independent hydraulic jack. This system follows the philosophy of the D-DIA but with the Kawai geometry. Our side station system is a small-scale prototype of this new guideblock. The Kawai

geometry has a long proven track record of being capable of providing higher pressure than the DIA on samples with the same volume.

B.1.5 Diamond Anvil Cell X-Ray Facility at the NSLS

Facility Overview

The diamond anvil cell X-ray (X17-DAC) facility at the National Synchrotron Light Source (NSLS) is located on a superconducting wiggler beamline and consists of two stations (X17C and X17B3) and a sample preparation/spectroscopy laboratory. The X17C beamline is a side station that runs 100% of the time, amounting to a maximum of 81 days for each of the three cycles during the year. The X17B3 beamline operates 33% of the time in dedicated mode with an additional 33% in shared mode when the X17B2 (multi-anvil) station is running. This nominally provides a maximum of 54 days per cycle. Both X17C and X17B3 beamline are available for energy dispersive (EDXD) and monochromatic (ADXD) x-ray diffraction experiments.

The X17 beamlines are NSLS Facility Beamlines with a Contributing User (CU) agreement with COMPRES. The NSLS is responsible for the operation of the beamline (optics, safety systems, etc.) while COMPRES is responsible for operation of the experimental stations. 50% of the beamtime is given to general users (GU) and 50% of the available beamtime (CU time) is assigned to COMPRES. All proposals are first submitted through the general proposal system at NSLS to compete for GU time. CU time may be assigned to proposals without a sufficiently high rating to obtain GU time, to increase the number of days for a successful GU proposal, or for use by beamline staff.

From 2007-2011, a multi-institution management team has led X17-DAC. The management team is headed by PIs Donald Weidner (Stony Brook) and Thomas Duffy (Princeton). The other members of the management team are: Mark Rivers (Chicago), Lars Ehm (NSLS/SBU) Alex Goncharov (Carnegie), Jiuhua Chen (FIU), and Sanjit Ghose (NSLS-II). Our staff consists of two beamline scientists: Xinguo Hong (since June 2010) and Zhiqiang Chen (since Sept. 2008).

Science Overview

From Jan. 2009-April 2011, X17C had more than 204 person-visits representing 44 separate universities and institutes while X17B3 had 57 person-visits representing 18 separate universities and institutes. Details on the number of proposals, beamtime assignments, and total usage are provided in the Appendix.

From January 2006 – April 2011, 184 publications (peer-reviewed journal and conference publications as well as theses) resulting from work at X17C and X17B3 have been recorded. The research capabilities of the NSLS DAC program are best illustrated by a few selected research highlights that are included below.

<u>Nanoscale Manipulation of the Properties of Solids at High Pressure with Relativistic Heavy Ions</u>

Zirconate pyrochlore, $Gd_2Zr_2O_7$, pressurized in a diamond-anvil cell (DAC) up to 40 GPa was irradiated at one of the world's largest ion accelerator facilities (GSI Helmholtz Center) with energetic uranium and xenon ions with kinetic energy of 45 GeV. The high velocity (~50% speed of light) of the ion beam allows traversal of mm-thick diamond anvils of the DAC before reaching the pressurized sample. As the projectiles lose kinetic energy along their trajectories, they induce intense electronic excitations and ionizations, which trigger complex processes within the solid. *In situ* synchrotron X-ray diffraction experiments (COMPRES beamline X17C, NSLS) on irradiated and non-irradiated reference samples revealed that a previously unquenchable, pyrochlore high pressure phase was recovered to ambient pressure after ion-beam exposure. Transmission electron microscopy evidenced a radiation-

induced, nano-crystalline texture. Quantum-mechanical calculations confirm that the surface energy at the nanoscale is the cause of the remarkable stabilization of the high-pressure phase. This work highlights the combined use of high-pressure and high-energy ion irradiation as a new strategy for manipulating and stabilizing novel materials to ambient conditions that otherwise could not be recovered.
M. Lang, F.X. Zhang, J.M. Zhang, J.W. Wang, B. Schuster, C. Trautmann, R. Neumann, U. Becker, R.C. Ewing, *Nature Materials* 8 (2009) 793-797.

Evidence of tetragonal nanodomains in the high-pressure polymorph of $BaTiO_3$

Perovskites play an important role in Earth's interior and in many industrial applications. A rich variety of disorder and nanostructures, which have an important influence on the elastic and electronic properties, have been reported in perovskites. The pressure induced P4mm→Pm-3m phase transition in the prototype perovskite $BaTiO_3$ was investigated by x-ray total scattering at the COMPRES supported beamline X17B3 at the National Synchrotron Light Source. The evolution of the structure was analyzed by fitting pair distribution functions over a pressure range from ambient pressure up to 6.85(7) GPa. Evidence for the existence of tetragonal ferroelectric nanodomains at high pressure was found. The average size of the nanodomains in the high-pressure phase decreases with increasing pressure. Extrapolation of the domain size to pressures higher than studied experimentally suggests a disappearance of the ferroelectric domains at about 9.3(5) GPa and a cubic symmetry of $BaTiO_3$ high-pressure phase. This work shows that pressure induced phase transitions can be very complex, with competing mechanisms on different length scales.
L. Ehm, L.A. Borkowski, J.B. Parise, S. Ghose, and Z.Chen, (2011) *Applied Physics Letters 98*, 021901.

Deformation of the Lower-Mantle Ferropericlase across the Electronic Spin Transition

Recent high-pressure studies have shown that an electronic spin transition of iron in ferropericlase, an expected major phase of Earth's lower mantle, results in changes in its properties, including density, incompressibility, radiative thermal conductivity, electrical conductivity, and sound velocities. To understand the rheology of ferropericlase across the spin transition, we have used in situ radial X-ray diffraction techniques to examine ferropericlase, $(Mg_{0.83},Fe_{0.17})O$, deformed non-hydrostatically in a diamond cell up to 81 GPa at room temperature. Compared with recent quasi-hydrostatic studies, the range of the spin transition is shifted by approximately 20 GPa as a result of the presence of large differential stress in the sample. We also observed a reduction in incompressibility and in the unit cell volume of 3% across the spin transition. Our radial X-ray diffraction results show that the {001} texture is the dominant lattice preferred orientation in ferropericlase across the spin transition and in the low-spin state. Viscoplastic self-consistent polycrystal plasticity simulations suggest that this preferred orientation pattern is produced by {1 1 0}<1−10> slip.
Lin, J., H. Wenk, M. Voltolini, S. Speziale, J. Shu, and T. Duffy (2009), Deformation of lower-mantle ferropericlase (Mg,Fe)O across the electronic spin transition, *Physics and Chemistry of Minerals*, *36*(10), 585-592.

Ionic high-pressure form of elemental boron

Boron is an element of fascinating chemical complexity. Although we now know of at least 16 polymorphs, the stable phase of boron is not yet experimentally established even at ambient conditions. Boron's complexities arise from frustration: situated between metals and insulators in the periodic table, boron has only three valence electrons, which would favor metallicity, but they are sufficiently localized that insulating states emerge. However, this subtle balance between metallic and insulating states is easily shifted by pressure, temperature and impurities.

The results of high-pressure experiments and *ab initio* evolutionary crystal structure predictions have found a new boron phase that we named γ-B_{28}. *In situ* high-pressure x-ray diffraction was performed at the X17C beamline of the NSLS.

The new phase is stable between 19 and 89 GPa, can be quenched to ambient conditions, and has a hitherto unknown structure (space group Pnnm, 28 atoms in the unit cell) consisting of icosahedral B-12 clusters and B-2 pairs in a NaCl- type arrangement. We find that the ionicity of the phase affects its electronic bandgap, infrared absorption and dielectric constants, and that it arises from the different electronic properties of the B-2 pairs and B-12 clusters and the resultant charge transfer between them.

Oganov, A. R., J. Chen, C. Gatti, Y. Ma, Y. Ma, C. W. Glass, Z. Liu, T. Yu, O. O. Kurakevych, and V. L. Solozhenko (2009), Ionic high-pressure form of elemental boron, *Nature*, *460*(7252), 292.

Thermal Expansion of Iron-Rich Alloys and Implications for the Earth's Core

The Earth's core makes up nearly one-third of the planet's mass. Its composition, properties, and dynamics are fundamental issues in the study of the Earth's interior. A critical test for any candidate core composition model is that it must be able to reproduce the physical properties of the core. On the basis of observing seismic rays penetrating the deep interior of the Earth and the orbital dynamics of the Earth as a planet in the solar system, models have been constructed to describe the physical state, density profile, and velocity profiles of the Earth's interior. One of the most widely used models is the Preliminary Reference Earth Model (PREM). To perform the test of consistency between a composition model and the PREM model, we must know the thermal state of the core and the equation-of-state (EOS) of various Fe-rich alloys at the pressure and temperature conditions of the core. A survey of literature revealed a significant lack of thermal expansion data on Fe-rich alloys under static high pressure. In this study, we have determined thermal expansion of Fe_3S, Fe, and Fe–Si alloys using in-situ synchrotron techniques. We have determined the unit-cell parameters and thermal expansivity of the iron–sulfur compound Fe_3S by using synchrotron x-ray diffraction techniques and externally heated diamond–anvil cells at pressures up to 42.5 GPa and temperatures up to 900 K. Our data at 42.5 GPa and 900 K suggest that 2.1 atom % sulfur produces 1% density deficit in iron.

B Chen, L Gao, K Funakoshi, J Li, Thermal Expansion of Iron-Rich Alloys and Implications for the Earth's Core, *Proc Natl Acad Sci USA*, 104: 9162-9167 (2007)

Single-Crystal X-Ray Diffraction and Brillouin Scattering of Hydrous Olivine Polymorphs

Brillouin scattering together with x-ray diffraction measurements at X17C, NSLS were used to constrain the anisotropic elastic properties of hydrous olivine and wadsleyite at high pressure. To determine single-crystal elastic constants, Brillouin spectroscopic data must be combined with constraints on density and crystal orientation. For each sample, we determine the unit cell volume and orientation of the single crystal using energy-dispersive x-ray diffraction techniques at X17C. Wadsleyite, β-Mg_2SiO_4, is potentially a major hydrogen host in the Earth's transition zone (410-660 km depth) due to its large water solubility. Determination of the effect of water on the elasticity of wadsleyite can provide constraints on the water content in the earth's transition zone through comparison with seismic data. In our first study [Mao et al., 2008], we showed that the elasticity of wadsleyite decreases strongly with increasing water content at ambient conditions. More recently, we have measured the single-crystal elastic properties of forsterite with 0.9 wt.% H_2O and wadsleyite, β-Mg2SiO4, with 0.84 wt.% H_2O to 12-14 GPa [Mao et al., 2009; 2010]. Aggregate bulk and shear moduli of hydrous forsterite increase with pressure at a greater rate than those of the corresponding anhydrous phase. Whereas V_P and V_S of hydrous forsterite are ~0.5% slower than those of anhydrous forsterite at ambient pressure, velocity crossovers at ~3–4 GPa result in higher velocities in hydrous forsterite at pressures corresponding to depths below ~120 km. At the pressure of the 410-km discontinuity, V_P and V_S of hydrous forsterite exceed those of anhydrous forsterite by 1-2%. This surprising finding has implications for the interpretation of seismic structure in potentially hydrogen-rich regions of the deep upper mantle.

Mao, Z., S. Jacobsen, F. Jiang, J. Smyth, C. Holl, D. Frost, and T. Duffy (2010), Velocity crossover between hydrous and anhydrous forsterite at high pressures, *Earth and Planetary Science Letters*, *293*(3-4), 250-258.

Mao, Z., S. D. Jacobsen, F. Jiang, J. R. Smyth, C. M. Holl, and T. S. Duffy (2008), Elasticity of hydrous wadsleyite to 12 GPa: Implications for Earth's transition zone, *Geophysical Research Letters*, *35*, L21305.

Mao, Z., S. Jacobsen, F. Jiang, J. Smyth, C. Holl, D. Frost, T. Duffy, Single-crystal elasticity of wadsleyites, ß-Mg_2SiO_4, containing 0.37-1.66 wt.% H_2O, *Earth Planet Sci. Lett.*, **268**, 540-549 (2008).

Technical Developments 2007-2011

Over the last 5 years, we have undertaken a comprehensive effort to enhance our infrastructure and upgrade many components throughout the facility that had become worn-out with age or outmoded. Both beamlines as well as the support laboratory have been extensively modified resulting in major enhancements in experimental quality and user experience.

X17C

At X17C, we conduct angle **and** energy dispersive x-ray diffraction on polycrystalline samples in either axial or radial geometry. Energy dispersive single-crystal diffraction techniques for phase identification, orientation determination, and unit cell refinement are also available. Experiments can be carried out at room temperature or at high temperatures (up to 800 C) using various external heating techniques such as user-supplied hydrothermal diamond anvil cells.

A new area detector (Rayonix SX-165) was acquired and has been in use since June 2009. New EPICS control of the MARCCD was installed to control and manage data collection remotely with fully automatic scan protocol.

Angle dispersive radial x-ray diffraction in a radial geometry was established and is now carried out routinely. A panoramic diamond anvil cell, available to users, was obtained for the beamline.

The sample stages at X17C, X17B3, and the ruby spectroscopy system were improved with a series of new components that replaced worn-out stages, motors, and mounts. We are in the process of installing new clean-up slit systems at both beamlines that will substantially reduce gasket contamination signals.

There have been several other developments. A new beamline control computer was set-up in 2010 and beamline software and electronics have been fully upgraded and are compatible with the latest versions of EPICS beamline control software. The performance of the double Laue monochromator at the beamline has been improved with the assistance of Z. Zhong (NSLS) and improved X-ray wavelength calibration procedures were instituted.

X17B3

X17B3 supports experiments at two x-ray energies: ~30 keV and ~80 keV. In the lower energy range, angle and energy dispersive x-ray diffraction experiments are carried out, similar to the capabilities at X17C. In addition to external heating, a laser heating system is the commissioning stage and will be available for users from the third cycle of 2011 onward.

Total X-Ray Scattering at 80 keV

At 80 keV, total scattering pair distribution function (PDF) measurements are performed. This is a novel technique that involves measuring both Bragg and diffuse scattering for structural analysis of complex materials, allowing determination of local atomic structure for both crystalline and amorphous materials. A recent paper by Ehm et al. (2011) demonstrates the capability of the technique. The number of user proposals for PDF experiments at X17B3 has increased substantially. This method can be used to study the structural properties of liquids, glasses, melts, nanomaterials, and disordered crystals. Geophysical applications include the study of structural evolution of melts and glasses with pressure and order-disorder phenomena under mantle conditions.

The detector capabilities of X17B3 were upgraded with the purchase of a novel amorphous Si flat panel detector (Perkin Elmer XRD 1621). This is a high-energy digital imaging detector with 15 Hz frame rate optimized for use at ~80 keV. It also features exceptional dynamic range and point spread function. A computer with special specifications to accommodate fast and high volume data was also obtained and EPICS controls established. The detector has been extensively tested and is working well for experiments at both 30 keV and 80 keV. A universal detector stage was also designed and installed at X17B3 to accommodate all types of detectors in use.

Laser Heating System

A double-side laser heating system for diamond anvil cell experiments has been designed, setup, and tested. The system uses a 100 W fiber laser. The compact system is set-up on a small 2' x 3' optical breadboard. Temperature measurements from both sides can be made using a newly purchased spectrometer and CCD detector (Princeton Instruments). The laser heated diamond anvil cell has wide applications in geoscience and planetary science, and the reintroduction of this capability with advanced capabilities opens up new regimes for geophysical experiments at NSLS and NSLS-II.

Other Improvements

A high-energy beam transport and shielding system was designed and installed resulting in dramatically reduced background scattering for all experiments. The Kirkpatrick-Baez focusing mirror system at X17B3 was improved with a new inert-gas enclosure to provide better long-term performance and extended mirror lifetime. The capability of micro-focusing the X-ray beam to ~5-10 um is crucial for high-pressure geophysics experiments providing higher beam flux and better spatial resolution for heating experiments and when pressure gradients are present.

High-Pressure Diamond Anvil Cell Laboratory

Our high-pressure sample preparation laboratory houses equipment for DAC sample preparation and includes a micro EDM (electric discharge machine) system, mechanical microdrill, high-resolution optical microscopes, electrical work station, mechanical work bench, sample loading tools and DAC tools, standard samples, pressure media and pressure indicators. A cryogenic gas loading system is available (nitrogen and argon) as well as the capability to obtain gas loading from the GSECARS/COMPRES system at APS via a mail-in service.

Our sample preparation lab and ruby spectroscopy system are used annually by more than 100 users from six or more beamlines (X17C, X17B3, U2A, X14A, X27A and X7A). We have completely revamped this laboratory and all major equipment is now updated. We have a wide selection of diamond anvil cells available for beamline staff and users including symmetric and panoramic cells as well as an Almax plate DAC. Recently obtained sample preparation equipment includes a Hylozoic EDM machine for drilling gasket holes and a Leica (M165) stereomicroscope replacing an outdated 15 year old microscope. Our ruby spectrometer was upgraded in 2010 with the installation of a 250-mW, 532-nm laser (Ventus, Laser Quantum) along with a new 0.5 meter spectrometer and CCD detector (Princeton Instruments).

Outreach and Community Activities

A few highlights of outreach activities are mentioned here. Former beamline scientist, Dr. Jingzhu Hu was awarded the 2008 NSLS Community Service Award in recognition of her 18 years of service to the high-pressure synchrotron community at NSLS. In 2010, a redesigned web page for X17-DAC went on-line. We have conducted a number of workshops over the last 5 years. Recently, L. Ehm co-organized a workshop on dynamic compression under extreme conditions at the 2011 NSLS users' meeting. A hands-on workshop to introduce students and new users to the capabilities of high-pressure facilities at NSLS is also being organized for 2011. A special issue of *Journal of Synchrotron Radiation* based on our NSLS workshop on "Advances in High-Pressure Science Using Synchrotron X-rays" was published in November 2009.

Technical Developments 2012-2014

The development program at X17-DAC will continue in the period from 2012-2014. We expect to maintain an active, productive user program through the end of operations at the NSLS. We will concentrate on improvement of technical capabilities that benefit current users but are also transferable to NSLS-II. Methodology developments will be coordinated with the proposed COMPRES Technology Officer (CTO) at the COMPRES Technology Center at Brookhaven National Laboratory (CTCB).

High Energy X-Ray Mirror Development

A new multi-layer lateral-gradient mirror for high-energy DAC experiments is being developed for X17B3 in collaboration with Sanjit Ghose (NSLS-II). The goal is to provide the first capability in the world to focus 75 keV X-rays for high-pressure experiments. This will create a unique capability for high Q experiments at high pressure, with unprecedented resolution in real space and will provide the users community at X17B3 with unique opportunities to study the atomic structure and its evolution of geologically important melts, glasses, and crystals at simultaneous high pressure and temperature relevant to Earths' interior. Eventually, an energy tunable monochromator will be installed at X17B3. The project serves as a test for NSLS-II development and the mirrors are being developed at the NSLS-II fabrication facility. The new multi-layer optics will have the same size as the currently use Pt-coated mirrors and therefore allow the use of the same mirror bender configuration as in our current Kirkpatrick-Baez (KB) mirrors.

Angle Dispersive Single Crystal Diffraction

Software for single-crystal x-ray diffraction in angle dispersive geometry will be set-up for use at both beamlines. This will provide an important new capability to our user community at minimal cost. Our goniometer at X17B3 has a detector rotation arm which gives it a mjaor advantage compared to single-crystal diffraction set-ups at other high-pressure beamlines in the U.S. as the ability to rotate the detector about the sample center results in dramatically improved peak resolution at high angles.

Single-crystal diffraction at high pressure is a rapidly growing technique that can be used to determine the crystal structure of high-pressure phases which often cannot be solved using polycrystalline data. The method also allows for accurate determination of the high-pressure structural response to compression and equation of state.. This method will be transferred to NSLS-II where single crystal diffraction is expected to play a bigger role due to the high spatial resolution of the beam, allowing smaller single crystals to be selected for analysis.

Other Developments

We will expand our in-house capabilities for compression experiments by acquiring additional diamond anvil cells including additional panoramic and plate type cells to support a greater diversity of experimental projects. Panoramic diamond cells provide wide angular access and are essential for experiments in a radial geometry (for studies of strength and deformation mechanisms) and inelastic scattering experiments (for sound velocities, elastic constants, lattice dynamics). Plate type cells together with Boehler-Almax anvils enable a wider x-ray aperture at high pressures (critical for single crystal and total scattering experiments).

We will increase the efficient use of beamtime and the throughput of users by obtaining a universal pneumatic (double-diaphragm membrane) device for controlling pressure. This system can be used with symmetric and most other types of diamond cells in common use. Membrane pressure control enables precise remote control of pressure without disturbing the sample allowing for more efficient data collection, very fine control of pressure changes, and allowing for densely spaced, accurate data collection in all types of experiments. Furthermore, in combination with resistive or laser heating, the use of membrane pressure control will allow for true isothermal experiments.

Our Leica stereomicroscope (M165) system will be upgraded with a camera system to provide a fast, real-time live video on a HD screen as well as image capture. In addition to providing high-quality imaging of experimental samples, this system is ideal for assisting and training novice users.

We will upgrade our ruby spectrometer with a new optics design to enable measurements of Raman spectra at high pressures. The combination of Raman spectroscopy and x-ray diffraction can provide novel insights into structure, bonding, and phase transitions at high pressures. These laboratory and spectroscopic upgrades will all be fully transferable to operations at NSLS-II discussed below.

For the laser heating system, we will obtain a new blackbody calibration source, glassy carbon mirrors, and beam shaper. Applications of laser heating will be a major emphasis over the next five year period as there is no other method that can reach the P-T conditions of Earth's deep mantle and core for study of phase transitions, equations of state, elastic properties, deformation behavior, melting, and phase relations.

We will adopt a new monochromator design for the beamline based on sagittal focusing of bent Laue crystals. This design has been developed and tested for NSLS and NSLS-II by Z. Zhong and provides a few hundred times increase in x-ray flux with good stability and tunability. We can readily adapt this design which is compatible with our KB mirror system for our existing beamlines, resulting in enhanced performance at modest cost.

B.1.6 COMPRES at the NSLS II

The NSLS-II is a state-of-the-art medium energy storage ring under construction at Brookhaven National Laboratory. The spectral coverage of the 3 GeV ring ranges from the far IR to hard x-rays and has up to ~10^4 times higher brightness than NSLS (up to 20 keV). Advanced x-ray optics will enable high spatial resolution (to 1 nm) and high energy resolution (0.1 meV) at specialized beamlines. The facility will accommodate 58 total beamlines including a large number (~27) with advanced insertion devices. The project is on schedule and within budget and the 50% construction milestone was passed in March 2011. Operations are scheduled to start in 2014 and it will be ready for scientific experiments in 2015.

The initial budget for the NSLS II only includes a small amount for constructing beamlines. At this point in time, through a proposal system, several beamlines have been identified for commissioning at the NSLS II. They fall into three categories: 1. Beamline is approved and funding is available, 2. Beamline is approved and funding is possible and a detailed conceptual design is being prepared, but funding is not definite, and 3. Beamline is approved and the NSLS II organization will work with the PI's to develop funding for the beamline.

COMPRES has been involved in promoting several beamlines which have been approved. These include XPD (X-ray Powder Diffraction) from category 1, 4DE (4-Dimensional Studies in Extreme Environments) from category 3, and FIS (Frontier Synchrotron Infrared Spectroscopy Beamline Under Extreme Conditions) from category 2, which have dedicated floor space for high pressure research, and TEC (Time-resolved X-ray Diffraction and Spectroscopy Under Extreme Conditions) from category 3.

XPD is a beamline dedicated to high resolution X-ray diffraction. Extreme environments are part of the agenda and COMPRES has formed a partnership with this beamline to provide high-pressure equipment and staff for high pressure studies. There is sufficient real estate to house both the multi – anvil and the diamond anvil facility in the D hutch of this beamline. In the initial phase, there will be only one x-ray beam that will be time shared by all programs using this beamline. Phase two will include a second beam that can run simultaneously, doubling the available of beam time. XPD will operate on a damping wiggler (NSLS-II DW90 in figure 3). Beamtime will be allocated by evaluation of general-user proposals. The quality of science will be the driving criterion for the allocations. The configuration of this beamline will be very similar to 13 ID-D at GSECARS where X-ray diffraction shares the beam with high pressure. XPD should be operational on day 1 of the NSLS II. Thus, the user program will have a continuous home in the transition from NSLS to NSLS II. Of course, we expect that there will be some time lost simply due to the transition.

4DE, which has been approved by the NSLS II, is a more ambitious high-pressure facility. It will house three beams on a 3.5 Tessla superconducting wiggler. One will be dedicated to diamond anvil cell research and one for multi-anvil research. The third beam will be a monochromatic beam from a single bounce monochromator. It will be used for both multi-anvil and diamond anvil experiments. We will pursue funding for this beamline with the hope of delivering user based facilities near the beginning of the NSLS II.

Several beamlines in category 1 potentially offer a strong synergy between the COMPRES community and the beamline. We anticipate development of COMPRES programs at the Inelastic X-ray Scattering beamline (IXS), the Hard X-ray Nanoprobe (HXN), the full field X-ray imaging from micron to nanometer scales (FXI) beamline, and the Hard X-ray Resonant Scattering (HIX) beamline.

In late 2014, the X17 high-pressure program will transition to the NSLS-II. We are proposing a dual approach: 1) establishment of a dedicated high-pressure diffraction station at XPD, one of the six flagship project beamlines that are part of the NSLS-II construction project. 2) Build a state-of-the-art high-pressure support laboratory and support scientific staff who will enable forefront high-pressure research projects to be carried out at many beamlines around the ring through partner user proposals (PUPs) and other activities. This plan has the considerable advantage of allowing experiments to be carried out at the most appropriate beamline (e.g. highest energy resolution or smallest spatial resolution) rather than restricted to a dedicated beamline for which various compromises and tradeoffs in beamline capabilities must be made. Ultimately, we envision one or more fully dedicated high-pressure beamlines in addition to the above. The program outlined here will establish high-pressure as an important component of research at the NSLS-II from its inception and extend the forefront high-pressure geoscience research program active at NSLS for the next ~20 years.

High Pressure Program at XPD

XPD is a powder diffraction beamline and is one of the 6 project beamlines of the NSLS-II. The insertion device is a damping wiggler and will provide tunable energy in the range of 30-80 keV. The beamline will have unique features including fast readout rates and high angular resolution. It is designed to emphasize measurements in various environmental cells including diamond anvil cells.

At the mature stage of the project, there will be both a main branch and a side branch, each of which would be capable of supporting high-pressure experiments. The main branch has 3 in-line endstations: station A houses beamline optics; station C has a diffractometer that can accommodate various sample environments (including diamond anvil cells); station D can house various custom sample environments. Station B is a side station optimized to a fixed high energy (~70 keV) for total scattering experiments (including those at high pressure). Station B can operate concurrently with the C and D stations which time-share a variable-energy monochromatic beam. The beamline location and associated lab and office space for XPD are already assigned and the preliminary design report has been completed and approved. The project is on-track for commissioning to begin in 2014.

Through an agreement with NSLS-II, COMPRES will join the XPD beamline and establish dedicated LVP and DAC programs in station D. The large size of this station (~10 m) will enable LVP and DAC set-ups simultaneously. Secondary focusing optics will be used to provide a small beam size (~10 um) for DAC experiments. All user beamtime for XPD will be allocated by the general user proposal review system with science impact as the main criterion for acceptance. COMPRES will support technical and scientific staff with expertise in high-pressure research at XPD. COMPRES will have representatives on the beamline advisory team (BAT) which represents the high-pressure Earth science user community and works with the facility to define the scientific mission and technical scope of the beamline.

At XPD, COMPRES will install the hardware and support facilities for a diamond anvil x-ray diffraction station with on-line double-sided laser heating capabilities and *in situ* temperature measurements. This will include a diffractometer optimized for DAC samples and appropriate detectors (CCD, flat panel). We will establish an off-line high-pressure laboratory that will be available to all XPD users and include a range of diamond cell designs and specialized anvils, all necessary sample preparation equipment, capabilities for high-pressure and cryogenic gas loading, laser milling, and fluorescence and Raman spectroscopy. COMPRES will also install the 1000 ton press, complete with the differential DIA (DDIA) and T-25 modules. Area detectors, as is now used on the X17B2 side station, will be used for the monochromatic angle dispersive signal.

As part of XPD, high-pressure DAC experiments will be possible in stations B,C, and D and MAC experiments in station D. Station D will provide the capabilities of x-ray diffraction with the laser heating that are in strong demand by the user community. Station D will also provide the full suite of large-volume high-pressure experiments that are available now in X17B2 as described above. High-energy experiments in station B will enable us to enhance our program for high-resolution structural analysis of complex samples including silicate melts and glasses at high pressures using the x-ray total scattering and pair distribution analysis. As part of the XPD beamline, high-pressure capabilities will be immediately available to users on opening of the NSLS-II. There is overlap in scientific and technical agendas between the COMPRES community and that of XPD, which will foster collaborations and developments that would not arise otherwise. COMPRES participation in XPD will initially be for three years beginning in October 2014, covering the period of this proposal. After that time, a thorough review will define further directions.

New High-Pressure Opportunities at NSLS II

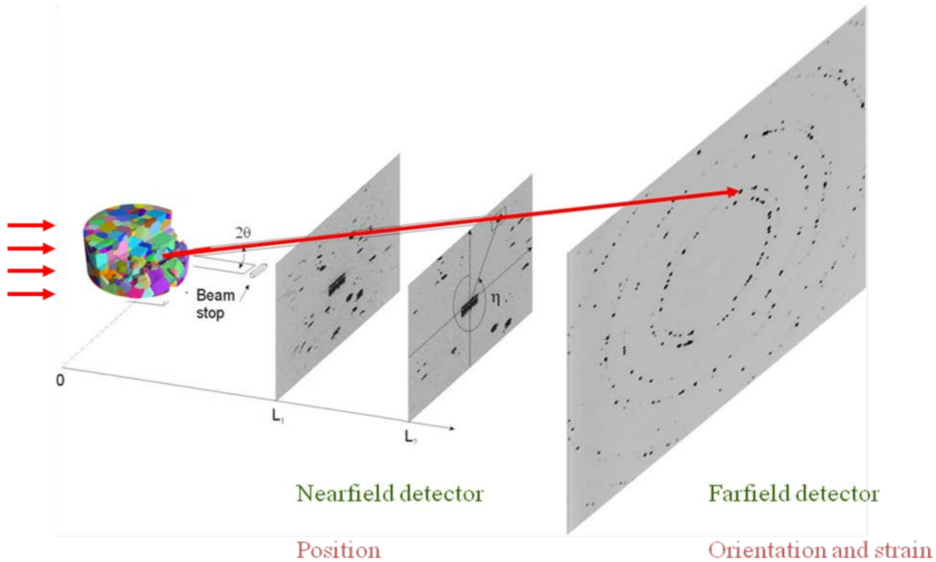
NSLS-II will provide new and exciting experimental capabilities for the COMPRES community. XPD along with other beamlines that have specialized capabilities will be cultivated for novel and forefront high-pressure experiments as described below:

Diffraction tomography

Grain interactions are extremely important in defining the properties of polycrystalline materials. Grain size dictates whether super-plastic flow, dislocations flow, or diffusion flow dominate the system evolution. As Ian Jackson's (ANU) program in Q measurements so well demonstrates, grain size dominates the Q of the samples that can be studied in high pressure systems. Furthermore, grain size is not an independent variable. During deformation grain size evolves attaining some equilibrium value appropriate to the stress – strain history. Thus, studies of time dependent mechanical properties need to allow grain sizes larger than those acceptable to standard X-ray powder diffraction methods. Otherwise, we are trapped in a parameter space that is not realistic and our ability to study grain size evolution is highly restricted. The Three Dimensional X-ray Diffraction Microscopy (3DXRD) technique can provide complete microstructural information of all grains within a volume containing up to 1,000's of individual grains (Oddershede et al., 2010; Poulsen, 2004; Poulsen et al., 2008; Wert et al., 2007; West et al., 2009). The sample is illuminated by a full field beam and 2D transmission diffraction images are taken using the rotation method. Using a large area far-field detector and reconstruction software, the full orientation, elastic strain tensor, volume, center of mass (5-10 microns), and unit cell refinement can be determined for every grain within the illuminated volume. In addition, by placing a high-resolution semi-transparent 2D or 3D near-field area detector close to the sample (~10mm) the 3D grain boundary morphology can be reconstructed using a back projection method. In this way the spatial and angular information are decoupled and more difficult samples (e.g., high mosaicity) can be reconstructed in extreme cases. For low mosaicity polycrystalline materials, however, the use of just a far field detector with the appropriate analysis software allows for time resolved studies of 100's – 1,000's of grains simultaneously following changes in orientation, strain, stoichiometry, and size as a function of applied forces (P/T). Although detailed local grain boundary information cannot be achieved without the use of a high-resolution detector, centers of mass can be determined along with volumes allowing for a tessellation process to determine the most likely grain neighbors. In this way, correlations between kinetics and local microstructural heterogeneities can be probed. The ability to collect grain-by-grain kinetics on a statistically significant number of grains rather than just measuring a powder-averaged value, will allow more realistic models to be developed that incorporate the true kinetic distributions of behavior due to the inhomogeneous nature of all polycrystalline materials.

Inelastic X-ray scattering beamline (IXS) and Hard Inelastic X-ray Scattering (HIX).

Inelastic X-ray scattering provides a powerful, high-resolution (~meV) technique for studying vibrational dynamics and electronic excitations at high pressures. For geophysics, the key capability is to measure the anistotropic elastic wave velocities as well as characterizing the detailed lattice vibrational spectrum. IXS experiments complement and extend more standard techniques such as Raman and Brillouin scattering at high pressure, overcoming limitations of optical experiments while providing unique information on phonon dispersion. Both single crystal and powder experiments at high pressures are possible. Applications such as X-ray Raman will also be feasible. X-ray Raman scattering is a medium-resolution (~1 meV) element-specific technique that provides similar information as XAS and EXAFS with the key advantage of not requiring very soft x-rays that cannot penetrate the DAC. The IXS beamline is designed to take advantage of the brightness and flux of the NSLS source by operating at ~10 keV and provide both medium (1 meV) and high (0.1 meV) resolution IXS capabilities, achieving an



order of magnitude improvement in resolution over existing beamlines around the world. Capability to accommodate DAC experiments has been built into the projects since inception (i.e., focusing to achieve ~1-10 um beamsizes).

One very exciting possibility to pursue on this beamline is the determination of the complete elastic tensor from high-pressure/temperature inelastic X-ray spectroscopy data of polycrystalline Earth materials. This would yield anisotropy information on powder samples. This is most relevant to the definition of paleo stress from seismic anisotropy. While measurements of this are in their infancy, this could be the goal of technique development.

The characteristic of the NSLS-II source is optimized for small intense beams in energy range from 2-25 keV. The brilliance of the source in this energy range is about three orders of magnitude higher compared to any other currently operating synchrotron radiation facility in the world. The impact on inelastic X-ray scattering experiments at extreme environmental conditions will be transformative. As part of the first suite of beamlines at NSLS-II, two inelastic scattering beamlines are being constructed. The characteristics are complementary; the inelastic scattering project beamline (IXS) will provide experimental capabilities with an unprecedented energy resolution of 0.1 meV, while the hard inelastic X-ray scattering (HIX) beamline is optimized for medium energy resolutions of less than 1 eV.

We plan to establish a formal collaboration between COMPRES researchers and the IXS and HIX beamlines in the form of a Partner User Proposal (PUP). The aim of the PUP will development of a pressure generating device on basis of the diamond anvil cell optimized for inelastic scattering at high pressure and temperature. Furthermore, the CTO will contribute in the further development of the collection and data analysis methodology. This development project will allow the COMPRES community to exploit an emerging experimental technique, which has a potentially transformative impact on our understanding of Earth's interior.

Nano-scale structural and chemical characterization of high pressure experiment product with novel synchrotron instrumentation

Earth materials show a large variety of heterogeneity, such as intergrowth, chemical disordering, element partitioning etc. While the *in situ* simultaneous determination of chemical and structural information at high pressure/temperature is technologically still out of reach, a combination of *in situ* high pressure synchrotron experiments with new state of the art synchrotron characterization methods, such as nanoprobes, will allow collection chemical and structural information on a nanometer scale on samples recovered from high pressure experiments.

In the project phase of NSLS-II two nanoprobe beamlines (HXN and SRX) will be constructer, which will allow the chemical and structural characterization of small heterogeneous samples with a spatial resolution of a around 10 nm. These beamlines would be ideally suited for the characterization of run products from high pressure/temperature experiments in small pressure generating devices such as diamond anvil cells.

SRX is designed for characterization of heterogenous materials and structures on micron to nanometer scales. Planned capabilities with potential novel DAC applications include: micro- X-ray absorption spectroscopy; x-ray emission spectroscopy; microdiffraction, and tomography with sub-micron resolution. Focusing capabilities operating up to 25 keV will provide a 100 nm sized beam with high flux compared to APS.

HRX is dedicated to providing high spatial resolution capability. Spatial resolution as low as 1 nm at 6-25 keV energy is the long-term goal. For high-pressure experiments, 10-100 nm beam sizes with adequate working distances for diamond anvil cell will be attained. Submicron beamsize and nano-diffraction capability is a frontier research direction for the high-pressure geosciences. Phases synthesized using the laser-heated DAC often have sub-micron dimensions. The beamline provide capabilities for analyzing very small single crystals, for selecting individual grains for study in a complex multi-component sample, and for miniaturization of the DAC to achieve much higher peak pressures.

We plan to establish a formal collaboration between COMPRES researchers and the SRX and HXN beamlines in a form of Partner User Proposal (PUP). The aim of the PUP will be to bring the most exciting high-profile mineral physics experiments to the nano-characterization facilities and contribute to the further development of the experimental methodology. The COMPRES lead development would mainly focus on the recovery of sample after high pressure/temperature experiments and the preparation of these heterogeneous samples (embedding, thinning etc) prior to the experiments at HXN and SRX.

Sample Fabrication and Characterization Capabilities

Our high-pressure technology center and team at NSLS-II (described below) will also develop a micro/nano fabrication facility and an advanced analytical facility taking advantage of the Center for Functional Nanomaterials (CFN) at BNL. The CFN is a user facility providing access to state-of-the-art capabilities including nanofabrication, lithography, dual beam SEM/FIB, deposition and etching, advanced electron microscopy, combined AFM/Raman instrumentation as well as synthesis and spectroscopy instrumentation. These resources offer important new capabilities for advanced sample preparation through specially designed and modified anvils and sample geometries as well as detailed chemical analysis of samples through extraction of recovered samples using the FIB technique together with high-resolution TEM analysis of composition and structure at the sub-micron level. The synthesis of high-resolution beamline capabilities, advanced analytical instrumentation, and staff with the expertise to advance the forefront of high-pressure technology and synchrotron techniques for geoscience applications is a unique combination that promises to make the COMPRES/NSLS-II program a worldwide leader in high-pressure Earth science and deep Earth geophysics research.

Operations Plan COMPTECH at NSLS-II

COMPRES resources will establish a team of scientists and technicians at Brookhaven National Laboratory, who will develop and promote synchrotron based geosciences research and be coordinated by a COMPRES Technology Officer (CTO). The objectives for the team would be:

- Assure successful experiments for COMPRES community members. This will emphasize proven techniques.
- Develop new experimental capabilities for beamlines at NSLS-II (e.g. next generation pressure tools, data evaluation tools etc). Collaboration with scientists in the community will be important to maintain a focused program that addresses fundamental Earth science questions.
- Conduct research at the forefront of geosciences utilizing NSLS-II and CFN experimental capabilities. The research team will develop personal research programs. This will help to keep them in touch with the pressing scientific issues.

- Operation of an outreach program which provides training and education to researchers of the geosciences community and develops new user communities in the field of Earths Sciences.

The COMPRES Technology Officer at Brookhaven National Laboratory will be a similar role as the COMPRES Technology Center at Argonne National Laboratory. The CTO will have three main goals: (i) Establish a successful technical developments programs (involving method and instrumentation development) focused on issues relevant to the COMPRES community, (ii) initiate collaborations with the lead scientists of NSLS-II beamlines currently under design/construction, which may have a relevance for the COMPRES community and explore solutions to integrate high-pressure Earth Sciences research into the beamlines research/user program, and (iii) establish a successful mineral physics centered research program that takes advantage of the existing capabilities at NSLS-II. Furthermore, the CTO will engage in fundraising efforts through spearheading proposals to funding agencies in order to supplement the Technology Center and grow the technical development and scientific program. Typically, proposals for funding will include the CTO along with scientists from the community that have a scientific stake in the success of the project. This keeps the science development project tuned to the scientific objectives and at the same time develops a group of users for the new technique once the development is complete.

The benefit for NSLS-II is an independent on-site center of expertise in high pressure geosciences. Furthermore, the envisioned technique and tool development of the team will add new and complementary capabilities to the existing experimental infrastructure at NSLS-II. The outreach and training effort will lead to (i) a broader user community for NSLS-II beamlines, (ii) a higher success rate for cutting-edge geosciences experiments due to well trained users, and (iii) contribute to development of the next generation of users and facility scientists.

Equipment Upgrades and Support Laboratory

Our current high-pressure equipment base has been significantly upgraded with COMPRES support and will allow us to establish a first-rate high-pressure program at NSLS-II immediately. This will be augmented with a number of equipment updates in the 2014-2017 period. The main equipment upgrades proposed for this period will be:

--table-top Kirkpatrick-Baez mirrors for XPD beamline providing ~10 um focal spot

--Gas loading system at NSLS-II. This system is essential for providing quasi-hydrostatic media and thermal insulation for laser heating.

-- On-line ruby spectroscopy system. In combination with diaphragm membrane pressure system, the time used for realignment and pressure changes will be drastically reduced

-- Portable laser heating system. We have already developed a compact design for laser heating, and we will modify to develop a modular, portable system that can be used at different beamlines.

We will be well positioned to tackle time resolved experiments at high pressure, opening a what is now a relatively unexplored field of *in situ* studies of kinetics of phase transitions, reactions and crystallization.

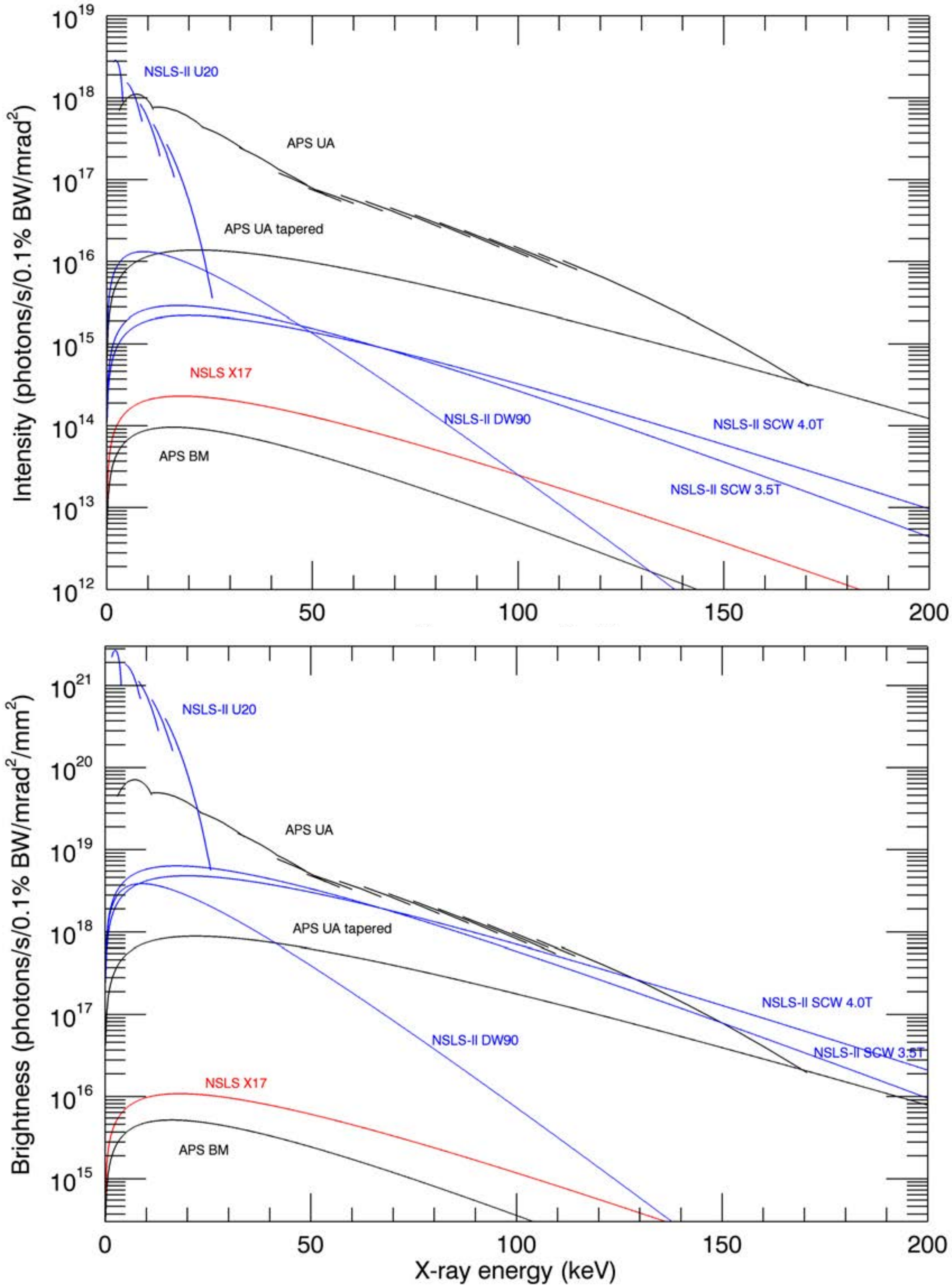


Figure 3. Brightness and Intensity of NSLS II beamlines compared with other beamlines.

Publications from the Multi-Anvil Program at Beamline X17B2

For the time period 2006 to July 2011, COMPRES support of beamline X17B2 resulted in 105 publications and graduate student theses. COMPRES support of beamline X17B3 and X17C resulted in 196 publications and theses. All papers and theses are listed in the complete list of COMPRES publications submitted as a supplementary document to this proposal.

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B.1.7 Infrared Diamond-Anvil High-Pressure Facilities at National Synchrotron Light Source (NSLS-U2A)

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

Progress Report for 2007-2011 and Proposed Plans for Operation and Development for 2012-2017

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Tremendous progress in the ultrahigh-pressure diamond-anvil cell experimentation made during in the past several decades has led to numerous breakthroughs in our understanding of the Earth. In many respects, these advances have been made possible by the coupling of diamond anvil cell and synchrotron techniques. This is particularly true for synchrotron based microspectroscopy under extreme conditions as diffraction limited spatial resolution is needed. The diamond anvil cell infrared beamline at the National Synchrotron Light Source (NSLS-U2A) is an integrated and dedicated facility for the measurement of far- to near-infrared spectra of materials from ambient to ultrahigh pressures at variable temperatures by coupling synchrotron infrared microspectroscopy, Raman scattering, and visible spectroscopy with diamond- and gem-anvil cell methods. Complementary and often unique information on the properties of Earth and planetary materials from near-surface conditions to those of the deepest interiors such as the bonding properties of crystals, glass, and melts can be obtained with these spectroscopic techniques. The presence of an IR beamline together with x-ray facilities for high-pressure experiments is one of the unique features of the NSLS for general users. We continue to broaden our user base, and provide convenient access for users from the COMPRES community. In addition, we promote our users' research projects on problems relating to high-pressure geoscience and planetary science, complemented by studies in materials science, condensed matter physics, chemistry, and biology (many of which are generated by the COMPRES community). The major beamline upgrades during the period of 2007-2011 have significantly improved the beamline performance and made the beamline operations more user-friendly. It also ensures that this unique facility is supplied/maintained with cutting-edge instrumentation.

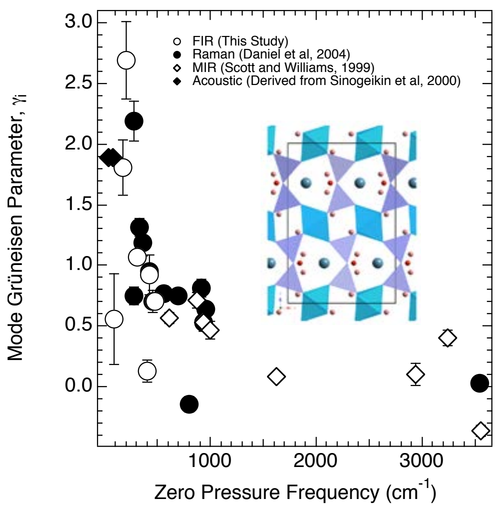
Science Highlights and Major Beamline Upgrades (June 1, 2007 to May 1, 2011)

Here we present the scientific highlights and technical advances at the beamline, focusing mainly on results of the past few years.

1. Synchrotron Infrared Spectroscopy of Hydrous Minerals

Infrared spectroscopy provides a key and oftenunique experimental approach with exceptional sensitivity to the O-H bonds and serves as an invaluable tool for evaluating the behavior of hydrous and nominally anhydrous minerals under extreme conditions. NSLS-U2A continues to provide all necessary tools for COMPRES user community to obtain insight into the optical properties of these minerals. Here are few highlights:

a. High-pressure infrared studies of talc and lawsonite



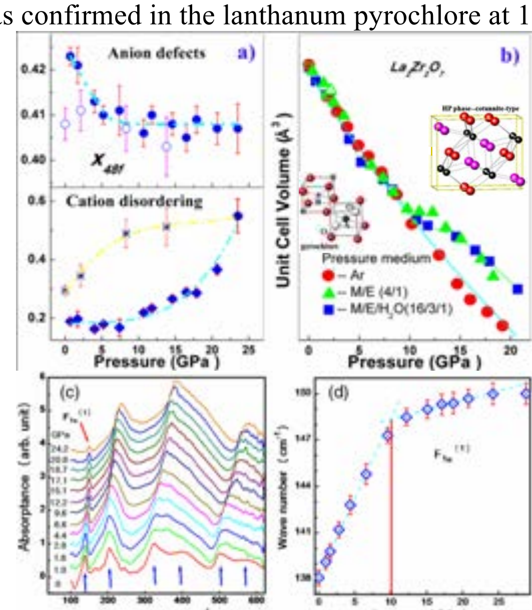
The high-pressure stability of hydrated metamorphic phases and their importance in transporting water into Earth's deep interior is well appreciated. Yet, the manner in which hydrogen is retained within these phases is a topic of considerable uncertainty. In particular, the role of hydrogen bonding (and its pressure dependence) in stabilizing these metamorphic phases at high pressure remains unclear. Among metamorphic phases stable to high pressures, talc ($Mg_3Si_4O_{10}(OH)_2$) and lawsonite ($CaAl_2Si_2O_7(OH)_2{\cdot}H_2O$) of state represent two different means of sequestering hydrogen. Talc is a chemically simple layer silicate, with the hydroxyl unit oriented nearly perpendicular to the layers and weak hydrogen bonding. In contrast, lawsonite contains both hydroxyl units and water molecules; it has both weak and intermediate strength hydrogen bonding. The effect of pressure on the vibrational spectrum of lawsonite has been examined by both mid-infrared spectroscopy and by Raman spectroscopy to ~20 GPa, but the far-infrared spectrum of lawsonite under pressure has not been reported. The vibrations in this long wavelength range are associated with translations of the water molecule and are critical for calculating the thermochemical properties of the material. In contrast, Raman spectra of talc have been Compilation of mode Grüneisen parameters versus zero pressure frequency obtained by the FIR measurements of the current study, MIR measurements from Scott and Williams (1999), Raman measurements from Daniel et al (2000) and acoustic values derived from Sinogeikin et al (2000). Note the strong frequency dependence of mode Grüneisen parameters; the low frequency modes provided by FIR are critically important to constrain the vibrational density
collected to only 3.5 GPa, and its infrared spectrum under pressure has not been reported. Synchrotron far-IR studies of talc and lawsonite have been performed at NSLS-U2A at high pressure and room temperature. For lawsonite, our data span the far infrared region from 150 to 550 cm^{-1} and have been extend to 25 GPa. The spectroscopic data were combined with previously published high-pressure mid-infrared and Raman data to constrain the Grüneisen parameter and vibrational density of states under pressure. In the case of talc, the high-pressure infrared data span both the mid and far-infrared from 150 to 3800 cm^{-1}, covering lattice, silicate, and hydroxyl stretching vibrations to 30 GPa. Both phases show remarkable metastability well beyond their nominal maximum thermodynamic stability at simultaneous high-pressure and high-temperature conditions [H. Scott et al., *Am. Mineral.* **92**, 1814-1820 (2007)].

b. Hydrogen bonds in the magnesium silicates, phase D and phase E

Hydrogen is a geochemically important element whose abundance is poorly constrained in the Earth's deep crust. The presence of hydrogen even in small quantities can affect phase relations, melting temperature, rheology, and other key properties of the deep Earth. Dense hydrous magnesium silicates (so-called alphabet phases) are a class of hydrous silicates that form under high *P-T* conditions in the $MgO–SiO_2–H_2O$. These silicates are potentially important components of the deep Earth water cycle, especially in cold slab environments. In particular, phase E is likely to be an important water carrier near the base of the upper mantle and transition zone, while phase D may be the major H_2O-bearing phase at the top of the lower mantle in low-temperature slab environments for peridotite compositions. Using the synchrotron infrared facility at NSLS-U2A, we measured the infrared spectra of polycrystalline samples of phases D and E up to 42 and 41 GPa, respectively. For both phases, at least three broad OH stretch vibrations were observed at elevated pressures indicating that each phase is comprised multiple hydrogen positions that exhibit disorder. No structural phase transition or amorphization was observed for either phase over the measured pressure range. The mode Grüneisen parameters of phases D and E are in the range of −0.12 to 1.14 and −0.17 to 0.83, respectively, with mean values of 0.41 (phase D) and 0.31 (phase E). Using empirical correlations of OH frequency and O· · ·H and O· · ·O bond lengths; the six OH vibrations of phase D at ambient pressure have corresponding O· · ·H and O· · ·O bond distances in the range of 1.519–1.946Å and 2.225–2.817Å, whereas the four OH vibrations of phase E have the corresponding O· · ·H and O· · ·O bond distances in the range of 1.572–2.693Å and 2.557–2.986 Å. These ranges encompass values reported from single-crystal x-ray diffraction measurements. At high pressures, the observable OH stretching vibrations exhibit both positive and negative pressure slopes. Our high-pressure infrared spectra for phase D do not support the occurrence of hydrogen symmetrization as predicted by first-principles calculations [S. Shieh et al., *Phys. Earth Planet. Inter*. **175**, 106-114, (2009)].

c. Pyrochlore under Pressure

Pyrochlore ($A_2B_2X_6Y$) is an isometric mineral structure with over 500 different compositions and a wide range of applications, from radiation resistant nuclear materials to being fast ionic conductors. Its properties change dramatically depending on composition and the degree of ordering on the cation sites and of the anion vacancies. However, the quantitative analysis of disordering at high pressure has always been a challenge. Researchers from the University of Michigan and the Geophysical Laboratory have demonstrated a new method for quantitatively measuring the degree of pressure-induced atomic disordering in pyrochlore oxides ($La_2Zr_2O_7$) using synchrotron x-ray diffraction (NSLS-X17C), synchrotron infrared spectroscopy (NSLS-U2A) and Raman scattering techniques. The research team, with the support of scientists from the diamond anvil cell (DAC) x-ray facility at the National Synchrotron Light Source (NSLS) have found that the disordering on the cation and anion sites has different influences on the individual diffraction peaks. Through careful analysis, their contributions to the diffraction intensities can be quantitatively distinguished. Using the Rietveld method, the research team demonstrated that anion disordering occurs first at pressures below 5 GPa and cation anti-site defects dominate above 10 GPa. An anomalous lattice expansion was confirmed in the lanthanum pyrochlore at 10 GPa by using x-ray diffraction, Raman

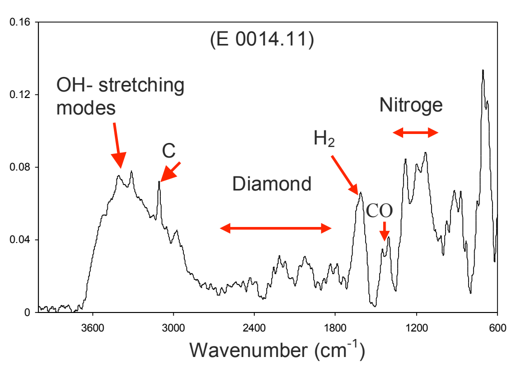


(a) Pressure dependence of x48f (pyrochlore structure) and degree of cation disordering with increase of pressure. The open symbols were measured during release of pressure. The pressure medium is methanol-ethanol-water; (b) Pressure (P)-volume (V) curves for La2Zr2O7 pyrochlore measured with different pressure media. The P-V curve of the case with Ar pressure medium can be well fit with the Brich-Murnaghan (B-M) equation of state. Open symbols were measured during release of pressure. (c) Synchrotron far-IR absorption spectra measured at various pressures; (d) Pressure dependence of the wave number of one sharp mode F1u also shows a discontinuity change in the slope.

scattering, and infrared absorption measurements in experiments where the pressure medium contained some water. In this study, such anomalous lattice expansion is attributed to the first report of the incorporation of water into the structure. Water intercalation in pyrochlore oxides may be common during the process of either pressurization or ion irradiation, and is mainly caused by the cation anti-site defects that result from disordering. In addition, $La_2Zr_2O_7$ is a catalyst which can split water during photon irradiation. The photochemical reaction may be closely related to the formation of anion disordering and the formation of Frenkel defects [F. X. Zhang et al., *Phys. Rev. Lett.* **105**, 015503 (2010)].

d. Water in microdiamonds from subduction zones: evidence from synchrotron-assisted infrared spectroscopy

All natural diamonds are formed in the Earth's deep interior – upper mantle, mantle transition zone, and perhaps below the 660 km seismic discontinuity. Due to the chemical inertness of diamond, it is a near-perfect container stable over geologically relevant times for fluid and solid inclusions trapped during its growth. The chemistry and structure of inclusions are used to reconstruct mantle mineralogy, conditions and compositions of diamond-forming media. Though most natural diamonds of gem and industrial quality originate from kimberlites and other ultramafic magmas, unusual small diamonds (1-300 micron in size) have been discovered within some orogenic belts of Kazakhstan, China, Norway, Germany, and Greece. All of them were formed as a result of Paleozoic and Mesozoic continental collisions and subduction of continental lithologies, followed by exhumation from minimum depths of ~150-250 km. Because these microdiamonds are hosted by metasedimentary rocks of continental affinity, their formation is unexpected according to mainstream geological thinking. They are thus currently the focus of a study to understand subduction zone processes and rock exhumation. A group led by L. Dobrzhinetskaya from the University of California at Riverside carried out synchrotron infrared microspectroscopy studies of samples from the Late Paleozoic crystalline massif of Erzgebirge at NSLS-U2A beamline. These studies showed that the Erzgebirge diamonds contain nitrogen impurities, molecular H_2O, OH-, and CO_3 radicals. The presence of both nitrogen C- and A defects classifies the studied diamonds as Type Ib-IaA, which is similar to other diamonds from metamorphic terranes such as the Kokchetav massif, Kazakhstan and the island of Fjortoft in the Western Gneiss region of Norway. Presence of H_2O and CO_3^{2-} strongly supports a concept of diamond crystallization from C-O-H fluids [L. Dobrzhinetskaya et al., *Earth Planet Sci. Lett.*, **248**, 325-334 (2006); *Proc Nat. Acad. Sci.* **104**, 9128-9132 (2007); *New Frontiers in* studies showed that the Erzgebirge diamonds contain nitrogen impurities, olecular H_2O, OH-, and CO_3 radicals. The presence of both nitrogen C- and A defects classifies the studied diamonds as Type Ib-IaA, which is similar to other diamonds from metamorphic terranes such as the Kokchetav massif, Kazakhstan and the island of Fjortoft in the Western Gneiss region of Norway. Presence of H_2O and CO_3^{2-} strongly supports a concept of diamond crystallization from C-O-H fluids [L. Dobrzhinetskaya et al., *Earth Planet Sci. Lett.*, **248**, 325-334 (2006); *Proc Nat. Acad. Sci.* **104**, 9128-9132 (2007); *New Frontiers in Integrated Solid Earth Sciences*, p. 373-395, Springer, New York (2010)]. The spectrum (E 0014.11) characteristics (cm^{-1}): OH stretching region: 3420-3320; Molecular H2O: 1620; CO3 radical: 1410; Nitrogen: A-defect – 1280; C-defect -1130 (1b), another C-defect at 1334 is not pronounced; Hydrogen (C-H):3110; Diamond phonon absorption bonds: 2600-2000



New IR/Raman Microscope System with Capability of Far-IR Reflection

The micro-Raman system at the U2A beamline is not only an important complementary tool with the synchrotron FTIR spectroscopy but also crucial for *in situ* pressure calibration at extreme conditions, *e.g.* diamond cell in cryostat or a resistive heating cell. An user-friendly infrared/Raman microscope system with the capacity of far-IR reflection with diffraction-limited spatial resolution has been built through the support of COMPRES and the Carnegie/DOE Alliance Center (CDAC). The new Raman/IR system has significantly improved the beamline performance. Here we present a few projects have been achieved on this system.

a. Infrared dielectric and vibrational properties of non-stoichiometric wüstite at high pressure: first high-pressure far-IR reflectivity studies at NSLS-U2A

Wüstite, Fe_xO, is a vacancy ridden mineral that crystallizes in the rocksalt structure. Due to its geophysical and technological importance, wüstite has attracted a wealth of theoretical and experimental investigations into its vibrational, dielectric and thermoelastic properties. Detailed theoretical investigations of wüstite often rely on comparison to thermoelastic data to corroborate their results. The far infrared (IR) reflectivity of $Fe_{0.91}O$ was investigated from 1 bar up to 33 GPa at room temperature using synchrotron FTIR reflectivity techniques in conjunction with the DAC at NSLS-U2A. The frequency of the fundamental transverse optic (TO) mode was found to be nearly independent of pressure up to 4.6 GPa followed by an increase of the TO frequency with pressure up to the rhombohedral phase transition. In addition, a second weak mode at 583 cm^{-1} and 1 bar was well resolved and found to shift to a higher frequency and increase in strength with pressure. This localized mode arises from the presence of vacancies in the crystal structure, and the relative strength of this mode suggests pressure induced charge localization near the vacancy sites. The data was fit with the classical Lorentz model with the addition of a plasmon resonance. This allowed an estimation of the electrical conductivity as well as plasmon-phonon coupling energies. The pressure dependencies of the dielectric properties of wüstite have been quantified, and their pressure derivatives show a change in sign near the pressure induced rhombohedral phase transition. Classical theories relating dielectric, vibrational and elastic properties are evaluated. In the case of the bulk modulus, the theory fails to reproduce accepted literature values [C. T. Seagle et al., *Phys. Rev. B*. **79**, 014104 (2009)].

b. Effects of pressure media on pressure-induced phase transitions in natrolite

Zeolites are hydrated framework alumosilicates which comprise an important class of low-density materials. Their frameworks are composed of corner connecting of TO_4 (Si, Al, Ge, Ga, *etc.*) tetrahedra which yield cavities and channels of molecular dimensions. Exchangeable non-framework cations occupy the cavities and channels along with absorbed water molecules at ambient conditions. As such, a plethora of structural studies have been carried out as a function of framework type, composition, and/or temperature in order to understand their relationship to catalytic, molecular sieving, and ion-exchange properties. In recent years, there has also been significant interest in pressure-induced structural and chemical changes in zeolites. Researchers from Yonsei University and Geophysical Laboratory have systematically studied the structural phase transitions in natrolite as a function of pressure and different hydrostatic media using micro-Raman scattering and synchrotron infrared (NSLS-U2A) spectroscopy. It was found that natrolite undergoes two reversible phase transitions at 0.86 and 1.53 GPa under pure water pressure medium. These phase transitions are characterized by the changes in the vibrational frequencies of four- and eight-membered rings related to the variations in the bridging T-O-T angles and the geometry of the elliptical eight-ring channels under pressure. Concomitant to the changes in the framework vibrational modes, the number of the O-H stretching vibrational modes of natrolite changes as a result of the rearrangements of the hydrogen bonds in the channels caused by a successive increase in the hydration level under hydrostatic pressure. Similar phase transitions were also observed at relatively higher pressures (1.13 and 1.59 GPa) under an alcohol-water pressure medium. Furthermore, no phase transition was found up to 2.52 GPa if a lower volume ratio of the alcohol-water to natrolite was employed. This indicates that the water content in the pressure media plays a crucial role in triggering the pressure-induced phase transitions in natrolite [D. Liu et al., *J. Phys. Chem. C* **114**, 18819–18824, (2010)].

c. Synchrotron infrared reflectivity of iron at high pressure

The physical properties of iron (Fe) at high-pressure place important constraints on the state and evolution of terrestrial planetary cores. Many techniques for measuring the physical properties of Fe at high pressures and temperatures rely on spectroradiometry for temperature measurement. Knowledge of the spectral emissivity of iron at high pressures is of immense significance for accurate temperature determination in laser-heated diamond anvil cells and dynamic shock wave experiments. It is commonly assumed that most materials are greybodies simply because emissivity data at high pressures do not exist. It is clear from measurements of the emissivity of Fe at 1 bar that iron is not a greybody, and almost no data exists for the optical properties of iron at high pressure. A group from the University of Chicago (C. Seagle and D. Heinz) measured the infrared reflectivity of iron at pressures up to 50 GPa and room temperature at NSLS-U2A using the FTIR Reflectivity technique in order to evaluate the greybody assumption often applied in spectroradiometry. The emissivity and other optical properties were derived with a Kramers-Kronig analysis. All the optical properties depend on pressure and undergo a discontinuous change in both slope and magnitude at the body centered cubic (bcc) to hexagonal close packed (hcp) phase transition in iron. The errors associated with the greybody assumption can be as high as 25% for bcc Fe assuming there is no temperature dependence of the emissivity; hcp Fe is nearly a perfect greybody at room temperature. The addition of high temperature data to this knowledge base will allow more accurate temperature determinations using spectroradiometry. The analysis of the high temperature data will also provide the pressure and temperature dependence of the conductivity, which is important for geodynamo simulations, and provide a fundamental understanding of the effect of pressure and temperature on the optical properties of Fe [C. T. Seagle et al., *Appl. Opt.* **48**, 545-552 (2009)].

Major Beamline Upgrades and Technical Developments

June 2009 to October 2010 was an exciting period in terms of major beamline upgrades and technical developments taking place at NSLS-U2A. The equipment funds supported by COMPRES, CDAC, and University of Nevada, Las Vegas (UNLV) became available in June 2009. This allowed the construction and operation of the new side station at NSLS-U2A to proceed on schedule. In addition, the COMPRES supplemental equipment funds boosted other beamline upgrades originally planned for the next few years. The detailed progress regarding these projects is described as below.

Construction, commission, and operation of the new side station at NSLS-U2A

High flux and high brightness synchrotron infrared (IR) radiations are required for high-pressure experiments using a DAC. However, the performance of an IR beamline is highly dependent on beam travel distance from the source spot to the end station. Diagnostic tests have shown that performance at the U2A beamline is significantly lower (by a factor of two) than the other IR beamlines of the NSLS in the mid-IR and much worse in the far-IR due to the U2A side station considerable distance (~15 meters) between the U2A end station (spectrometer/microscope) in the hutch and the synchrotron source. 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). Founded by COMPRES, CDAC, and UNLV, a new side station was constructed in December, 2009, and became operational in January 2010. The side station includes a Bruker Vertex 80v FTIR spectrometer and a Bruker Hyperion 2000 IR microscope ideally for the mapping of natural samples (*e.g.,* solid and fluid inclusions in thin section), heterogeneous charges from high-pressure experiments, as well as laser heated samples *in situ* at very high pressure in diamond anvil cells. The distance from the synchrotron source to the side station is only ~3 meters, which effectively eliminates the problems of beam divergence and image distortion. It is important to point out that all the developments on the side station project have been one in a very short period of time during the NSLS winter shutdown and commissioned the complete system right after the synchrotron beam came back in early January. As a result, no scheduled user beam time was affected due to the construction. Instead, many users (most of them from the COMPRES community) took the advantage of the side station as soon as it became available in January, 2010. Ongoing development at the side station is focused on building additional custom IR microscopes dedicated to far-IR experiments at high pressure or low-T and high-P experiments in the range from far- to near-IR. The side station project will be fully completed in 2011 with an onsite small ruby system and enclosure surrounding all the instruments on the optical table.

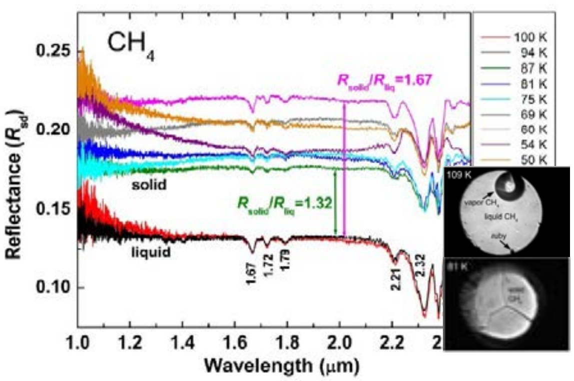
High-pressure and low-temperature IR/Raman capability with compact cryostat

The combined high-pressure and low-temperature techniques are very important to address a broad range of problems in planetary sciences. Such capabilities could also attract a lot of users. The new cryostat from Cryoindustries with a compact design for accommodating standard symmetric DACs was purchased with the COMPRES supplemental equipment funds and was delivered in February, 2010. The cryostat was immediately tested right away and the first user experiment took place on March 10, 2010 (user group from the Ohio State University). The experiments conducted with the cryostat demonstrate that the performance of the cryostat is well above the specifications and much more user friendly. Thus, the combined cryostat and standard symmetric DACs provide a way for general users to routinely and reliably perform *in situ* high pressure and low temperature IR studies at NSLS-U2A. The following is a highlight related to this technique.

Low-Temperature Infrared Reflectivity of CH_4: Application to Hydrocarbon Lakes on Titan

Methane is abundant on icy bodies in the solar system including Pluto and Triton, as identified in observational spectra from characteristic absorption bands at around 1.7 and 2.3 μm. The physical state of CH_4 and other hydrocarbons in the solar system, however, cannot simply be interpreted from the positions of absorption bands alone. On the other hand, reflectance spectroscopy provides one of the few direct observations of outer solar system bodies for probing their surface compositions. Recent discoveries of lakes on Titan, Saturn's largest moon, from the *Cassini* mission come from interpreting a compelling combination of low-lying and flat surfaces measured by Radar and corresponding dark features observed by infrared (IR) reflectivity. It was thought that the Titan's surface is near the triple point of methane and models of Titan's methane cycle are therefore analogous to the Earth's hydrologic cycle, where rain and clouds of methane exchange with solid and liquid methane on the surface. However, interpretation of recent and forthcoming mission observations of planetary bodies requires laboratory-based reflectance spectroscopy of planetary ices and liquids at relevant temperatures, especially to distinguish states of surface phases. Using intense synchrotron infrared radiation at NSLS-U2A, researchers from Northwesten University and the Geophysical Laboratory studied the reflectivity of solid and liquid CH_4 at temperatures from 50-100 K. At conditions below the critical pressure, co-existing states of vapor and liquid CH_4 were observed at temperatures down to 94 K. Upon crystallization below 94 K, a dramatic increase in reflectance of CH_4 was observed at the diamond-sample interface (R_{sd}). Whereas the position of characteristic absorption bands of CH_4 are insensitive to the phase state, darkening of liquid CH_4 in reflectance is consistent with Titan's observed dark surface features, which may represent large polar lakes forming seasonally through exchange with clouds and rain of methane where Titan's surface temperatures are within

IR reflectance of methane at the sample-diamond interface (*R*sd) as a function of temperature (top). Spectra obtained at 100 and 94 K are from the liquid phase and at 87 K and below the sample was solid CH4.



a few degrees of the triple point of methane [K. A. Adams et al, Visible and near infrared reflectivity of solid and liquid methane: application to hydrocarbon lakes on Titan, *to be published.*]

CO_2 laser heating system

The ability to generate high pressure and high temperature is crucial for the COMPRES user community to address a range of problems on mineral physics/chemistry related to the Earth's interior. High-pressure and temperature >1000 K are essential for infrared studies of Earth and planetary materials. CO_2 laser heating techniques combined with DACs are the best approach to creating such extreme conditions for studying the water solubility in lower mantle minerals. We continued to make progress on the CO_2 laser heating system during the period of 2009-2010. Major progress has been made on the laser interlock system, which been installed by the NSLS interlock group to comply with the laser safety requirements at BNL. In addition, an entirely new temperature calibration system including a spectrometer with an OMA-InGaAs detector from Princeton Instruments has been purchased with the COMPRES supplemental equipment funds and has been installed and fully tested. We will focus on the process of getting the safety operation procedure (SOP) approved by the BNL laser officer and move the whole system into the commissioning phase. This off-line laser heating system will be available to the COMPRES user community in the summer of 2011. Further development on the *in- situ* laser heating techniques combined with synchrotron IR spectroscopy will be the highest priority during the next phase of COMPRES as described below.

Scientific and Technical Challenges for the Future and a Vision for NSLS-II during the next phase of COMPRES (June 1, 2012 to May 31, 2017)

Water, stored in planetary materials as molecular H_2O or OH defects, has a great effect on their physical properties, including density, seismic velocities, anisotropy, strength, and viscosity. The remarkable ability of mantle materials to incorporate water at high pressures, investigated by largely infrared spectroscopy, has lead to a greater awareness of the potential role of water in shaping physical and geochemical evolution of the Earth. Water, among other volatiles such as CO_2 may control melting in the mantle, weakening of the lithosphere, and thus plate tectonics itself allowing our habitable planet to develop. The study of Earth's deep water cycle has become a central topic in solid-Earth geophysics and is recognized as a frontier area of research (see http://www.nap.edu/catalog.php?record_id=12161).

It is well known that a large amount of water is carried into the mantle by hydrous minerals on top of the oceanic lithosphere, but the amount of water carried into the deep mantle in high-pressure nominally anhydrous minerals (NAMs) remains an area of exploration. Extensive laboratory studies have been carried out to determine the solubility of water (hydrogen) in these minerals. By combining experimental studies and thermodynamic analysis, mechanisms of water dissolution (hydrogen dissolution) in some minerals are becoming evident through IR spectroscopy and other methods. In most cases, water (hydrogen) is dissolved in NAMs as hydroxyl point defects, and a better understanding of deep-mantle hydration at the atomic scale will lead to new breakthroughs in understanding the associated effects on physical properties and mantle dynamics. There is particular uncertainty in the ability of lower mantle minerals to incorporate H_2O required to better model global water circulation. Due to the small sample volumes, characterizing water (hydrogen) content in lower mantle minerals is challenging and the current discrepancies in H_2O content of deep mantle phases range from almost zero to thousands of parts per million.

Determining water solubility in lower mantle minerals is a key to understanding global water circulation. Among various volatile elements, hydrogen is unique in its ability to change the physical properties dramatically even at a small fraction. For example, even ~0.01 wt% of water can increase electrical conductivity by a factor of ~100. The reduction of seismic wave velocities by hydration of high-pressure minerals is on the order of effects caused by temperature changes of 300-600 C. Hydrogen also affects other properties such as seismic wave attenuation and seismic anisotropy. Consequently, water (hydrogen) content can be mapped using several geophysical (and geochemical) observations. Critical to such an approach is better characterization of the role of hydrogen and its effects on some of the physical and chemical properties of minerals. This requires careful analysis of the nature of hydrogen bonding in mantle minerals.

In the experimental characterization of water (hydrogen) in minerals through high *P-T* experiments, one of the technical issues is the difficulty in quenching water (hydrogen). In conventional experimental studies, one prepares a sample at high *P-T*, quenches it (in terms of pressure and temperature) and measures the water content at ambient conditions. In this method, one assumes that water (hydrogen) dissolved in a sample at high *P-T* remains at the same site or with the same speciation during temperature and pressure quenching. This assumption is not necessarily valid in view of the fast diffusion of hydrogen, and some natural samples from upper mantle show evidence for rapid dehydration on assent by high-spatial resolution IR-mapping of water concentrations. A synchrotron IR facility will afford the ability to map hydrogen diffusion profiles in both natural and synthetic samples from experiment allowing a better understanding of hydrogen mobility in the Earth.

A number of intriguing questions are raised if the mantle is a reservoir for potentially vast quantities of volatiles:

a) What are all the possible hydrous and nominally anhydrous phases in which water could be retained at depth? The answer to this question is of the most fundamental importance, and will involve phase equilibrium and saturation experiments over a broad range of pressures, temperatures, and compositions.

b) How is water distributed in Earth's interior and how does it cycle from deep interior of the Earth to the surface? Why has the ocean mass fluctuated so little in the past that allowed the evolution of life on Earth?

c) What subduction-zone minerals can retain water and carbon until they are in the transition zone where they can be readily absorbed by minerals there?

d) When mantle rocks rise above 410 km depth, do saturated minerals dehydrate, thus releasing dense aqueous melts that are recycled to depth at mantle downwellings? That is, is the 410 km seismic discontinuity a dehydration boundary or "water filter"?

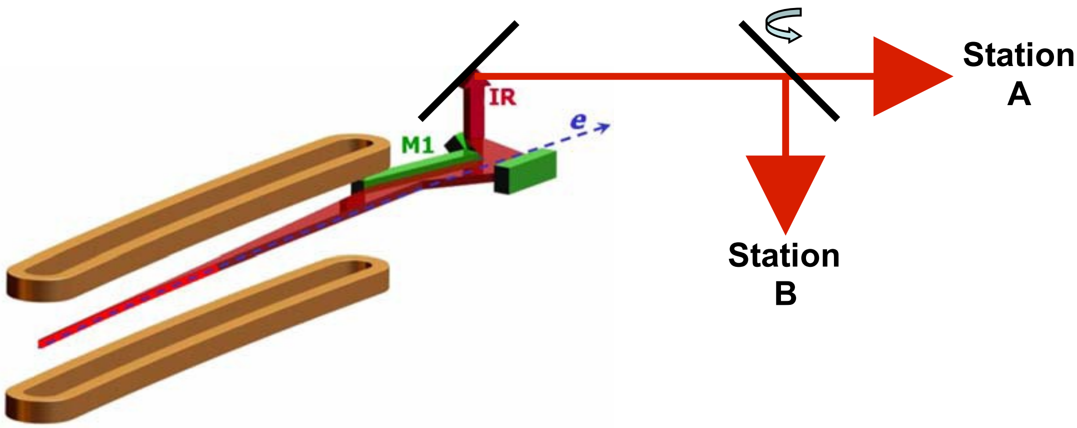
e) How do small amounts of water affect the rheologic properties of high-pressure minerals and the patterns of convection in the deep Earth?

f) How do small amounts of water affect the concentration (and mobility) of point defects and hence electrical conductivity?

Infrared spectroscopy provides a key and often unique experimental approach for addressing these problems due to its exceptional sensitivity to the O-H and C-O bonds and serves as an invaluable tool for evaluating the behavior of hydrogen and carbon at high *P-T* conditions as well as during cooling. However, it is essential to extend diamond anvil cell techniques to higher temperatures and pressures with either external heating or laser heating techniques in order to directly probe thermodynamics, chemical diffusion, and molecular coordination under high *P-T* conditions. This requires brighter sources with diffraction-limited performance in the infrared range and superior stability.

The National Synchrotron Light Source II (NSLS-II), a new synchrotron light source currently under construction at Brookhaven National Laboratory, will provide an ideal infrared source with the capacity of the large-gap dipole for such studies to couple the high *P-T* DAC techniques with the FTIR spectroscopic measurements and to address these scientific questions and challenges related to Earth and Planetary Sciences. Our proposal titled "Frontier Synchrotron Infrared Spectroscopy Beamline under Extreme Conditions (FIS)" has been approved by the NSLS-II Scientific Advisory Committee and the conceptual design is currently underway. The proposed high-pressure IR facility has a large-gap (90 mm) dipole infrared source and extraction port providing the full IR spectral range (including the far-infrared) to two experimental endstations. One of the endstations will be for the DAC or gem-anvil cell, and the other will be for the dynamic compression (such as gas-gun) apparatus. All equipment as well as the techniques currently available or under development will be utilized at the new facility at NSLS-II. The experimental capabilities of the two infrared endstations are complementary to other high pressure x-ray diffraction beamlines proposed for the NSLS-II.

Overall, the proposed high-pressure infrared beamline should produce spectral brightness equal to the world class performance of the NSLS in the far-IR region and up to an order of magnitude greater in the mid-IR range which is crucial for the *in-situ* high *P-T* IR studies on hydrous and nominally anhydrous minerals with combined DAC and laser heating techniques. In addition, the ring is designed for an extremely stable beam; an important characteristic for the standard rapid-scan interferometric techniques for infrared time-resolved applications. (Typically, the RMS pulse length will be in the range of 15-30 picoseconds and the time between bunches will be 2 nanoseconds).



Schematic infrared dipole radiation extraction: the bright red segment marks the electron beam segment serving as an infrared source. The radiation is collected by a long mirror (M1) and reflected out of the dipole chamber by a second and third mirror combination (not shown in the drawing). The collimated beam is delivered to station A or B with a switch mirror.

Station A – Diamond Anvil Cell Station

Station A will be an integrated and dedicated infrared beamline for experiments at simultaneous high pressure and variable temperature. The major experimental setup including the Bruker Vertex-80v FTIR spectrometer attached to a Hyperion-2000 IR microscope and other custom IR microscopes, a permanent CO_2 and fiber laser heating system, a compact cryostat with CVD diamond windows and other spectroscopic systems including micro-Raman. The station will be optimized for the following high-demand experimental techniques.

Infrared spectroscopy at high-pressure and ambient temperature

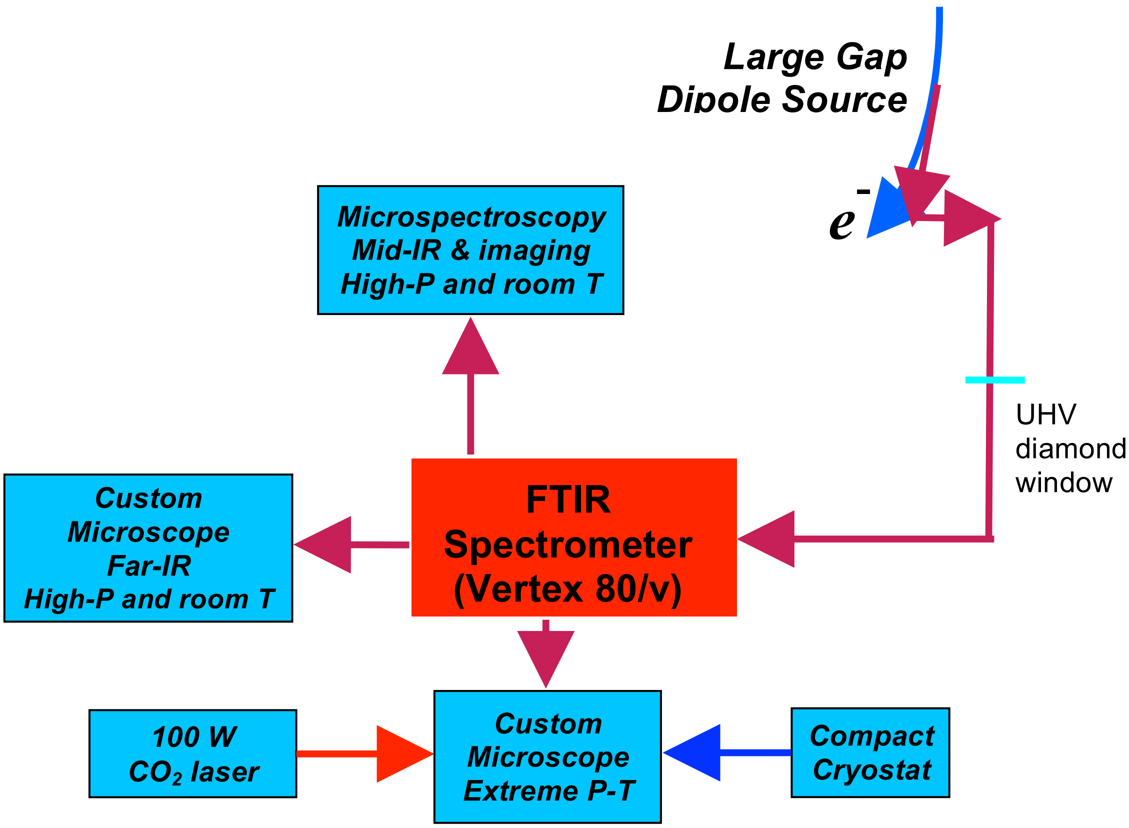
Measurements of phonon vibrations, phase transformation, band structures, and optical properties at high pressure.

In-situ laser heating infrared spectroscopy at high *P-T* conditions

Study hydrous and nominally anhydrous minerals under lower mantle conditions to gain insight into the incorporation of water (as hydroxyl) into the minerals. Synthesize and characterize new materials under high *P-T* conditions.

In-situ spectroscopy at high pressure and low temperature

Measurements of phonon vibrations, phase transformation and diagram, band structures, and optical properties at high pressure and low temperature with integrated techniques including synchrotron IR, Raman scattering, and visible-UV spectroscopy.



Schematic of the endstation A for high-pressure studies using DACs at variable temperatures. Three IR microscopes are attached to the FTIR spectrometer (vacuum bench).

Station B – Dynamic Measurement Station

Station B will be a multi-purpose station for dynamic measurements led by a team from Sandia National Laboratories. Impact experiments, using gas or powder gun launchers, will be installed as needed. Pulsed power systems, which utilize electromagnetic compression, will be considered in the future. Infrared/visible reflectance and/or transmission will be the primary diagnostics in the dynamic compression experiments at this station. Secondary diagnostics, such as laser interferometry, will be used to monitor the performance of the drivers. High-speed diagnostic development and testing may be performed at Station B when dynamic compression experiments are not underway. This station will be also made available for other dynamic measurements, such as transient laser heating.

We should emphasize that it is impossible to make these progresses without the support of the COMPRES user community. The continuing funding support through COMPRES is essential to ensure a smooth transition of the high-pressure IR program from NSLS to NSLS-II. In return, a most desirable IR facility for the entire COMPRES community will be available in early 2015 at NSLS-II.

B.2: Infrastructure Development Projects

B.2.1 Multi-Anvil Cell Assembly Development Project

Kurt Leinenweber, Thomas G. Sharp, James A. Tyburczy (Arizona State University)
Yanbin Wang (GSECARS, University of Chicago)

P.roposal for the new COMPRES: June 2012 – May 2017

Introduction

The multi-anvil cell assembly development project has existed as an infrastructure development project since the inception of COMPRES. The purpose of the project is to develop multi-anvil cell assemblies and then make them readily available to be used in any laboratory with a multi-anvil press. A series of cell assemblies of different sizes and pressure/temperature capabilities has been developed, using both preexisting and new materials and techniques. These cell assemblies involve many components with complex shapes and compositions, that need to fit together and function as a successful working high-pressure cell. All of these components – the pressure medium, gasketing, thermocouple, thermal insulation, furnace, and sample container – are specially made and are not generally available "off the shelf." This presents a requirement that a significant amount of development is necessary to achieve "first pressure" in a new laboratory starting from scratch. Our project has made it possible for many laboratories, both new and established, to achieve predefined pressure/temperature conditions readily, and to quickly set about pursuing the research that their laboratories were created for.

Since the beginning, a parallel purpose of the project has been to provide a way for users to readily obtain large volume high-pressure data on a synchrotron beam line. A series of cell assemblies that are based on the standard designs described above, but are modified to allow the passage of x-ray beams, have been simultaneously developed, and used for *in-situ* studies by groups who have applied for and received beam time on a synchrotron. The familiarity of the assemblies to those who use them offline makes these modified assemblies easy to use, allowing the researchers to focus on the data collection and interpretation of their beam line experiments.

We strongly believe that this project is providing a useful service to the high pressure community, and are seeking to continue this project in the new COMPRES. In the new mode, the standard assemblies will continue to be provided to the community, and the cost recovery from these assemblies will make that part of the project self-sustaining. Our funding request is for the development of new abilities and techniques, both in the area of new cell assemblies and capabilities, and in strengthening the overall quality and success of the existing assemblies. A stronger and more integrated focus on beam line development is enabled by the inclusion of Yanbin Wang, from the University of Chicago beam line GSECARS, as a new PI on the project. We will pursue further beam line developments with this beam line as well as beam line X17B at the National Synchrotron Light Source in Brookhaven. And we will continue to follow the needs of the community and the suggestions and advice of the COMPRES committees in formulating new ideas and in choosing new directions.

Cell Designs for Conventional Pressure/Temperature Experiments

The most widely distributed activity in the project, and probably the most visible to the general geoscience community, is the provision of multi-anvil cell designs for conventional multi-anvil synthesis and phase equilibrium experiments. We have a broad series of cell assemblies (Table 1), and they are in widespread use in various laboratories around the world (Table 2). A bibliography of twenty-two publications that have used the assemblies, arranged by year, is given in the Reference list.

In cases where the assemblies and their materials are well-established and people are using them on a continuing basis, the project will be made self-sustaining by the cost recovery from the assemblies. The COMPRES development will be focused on developing and improving the capabilities and characterization of the existing assemblies and on creating new designs.

One area of this need is higher temperature. It is inevitable, we have found, that research groups will desire assemblies that go beyond the pressure and temperature capabilities that exist – they naturally encounter the limits as they pursue their research objectives. One such area is in the achievement of higher temperatures. We currently lack higher temperature capabilities at lower pressures (pressures below 15 GPa and temperatures above 1200 °C to 1500 °C). We need to develop cell assemblies specifically to reach temperatures of 2000 °C and above in the lower pressure ranges. This effort was impeded recently by problems with the lanthanum chromite supply from Japan, but these problems have recently been solved with new formulations, and we will use COMPRES funding to use the new formulas and develop higher-T assemblies for lower pressure melting experiments, glass syntheses and others. The higher pressure 10/5 and 8/3 assemblies have much greater temperature capabilities already (in excess of 2200 °C) but even higher temperatures are still useful and a temperature limit of 3000 °C will be sought by eliminating melting components such as Al_2O_3 and the ZrO_2 cement from the assemblies. Another advantage of these extended temperature capabilities is that they will simultaneously represent a longer time capability at the current limit for those two assemblies, where the temperatures of 2200 °C and above are only achievable for a matter of minutes.

Another area is larger volume. The synthesis of large samples requires large-volume assemblies that are specialized for this purpose. We have developed 18/12 and 25/15 assemblies recently for this purpose, but they are not routine to fabricate yet and some development of ceramic techniques (extrusion of zirconia, porous mullite octahedra) are still needed to make an inexpensive off-the-shelf version that can be maintained by cost of sales at prices that are affordable to academic research laboratories.

The COMPRES development funding will be used to address the problem of "underperforming" assemblies. A current example is our G2 box furnace assembly, a Stony Brook design that has drifted downward in its pressure capabilities for unknown reasons. We suspect a change in the zirconia ceramic or some other systemic issue. In this regard, we need to note that in a project such as this, we need to be very realistic about the actual capabilities, success rates, and overall expectations of assemblies. It is one thing to claim a capability or success rate within your own laboratory, but quite another to provide assemblies to others and claim such capabilities, because the capabilities will be quickly brought to the test.

In order to improve our ability to monitor and characterize the assemblies, some funding is requested for monitoring equipment – we wish to log pressure, temperature, furnace voltage, furnace amperage, and other relevant information into a computer log for detailed analysis of the assembly behavior in real time. Other laboratories have these capabilities (Minnesota and GSECARS come to mind) but we would like to install them in-house in order to help with the design and troubleshooting of our assemblies. These capabilities will also be important for the beam line and all other assemblies described below. The budgeting for this equipment is described in the section on Capital improvements.

Improved multi-anvil cells for *in-situ* x-ray diffraction

For the past several years this project has been characterized by a strong and increasing interaction with beam lines, particularly the GSECARS beam line where many of the standard assemblies were calibrated, and where the new beam line versions were tested and used, in the large-volume press (LVP) at Sector 13, but also with beam line X17B at NSLS, where various DIA assemblies and materials were tested. Some of this work was presented in Leinenweber et al. (2006) and an update is given in Leinenweber et al., 2011, and eight of the other scientific publications in the reference list are primarily based on *in situ* x-ray measurements using our assemblies and materials. We now wish to go beyond our previous involvement and develop a truly optimized set of assemblies that are capable of very clear beam access to the sample as well as optimal pressure and temperature capabilities with high success rates. We intend to have a set of assemblies that are comparable in performance to the “planar” assemblies that are used in Japan, in which virtually the entire beam path is occupied by low-Z materials. In order to pursue this objective, we have included Yanbin Wang, from the GSECARS beam line, as a PI on the new version of this project.

Our current beam line assemblies are based on thermally insulating porous mullite pressure media (specially developed for our project) and either equatorial MgO windows (the 14/8 and 10/5 equatorial assemblies) or rhenium heaters with laser-cut windows (the 10/5 and 8/3 windowed assemblies). Recent re-designs of the laser-cut windows have allowed more open x-ray access. However, the x-ray beam still has to pass through pyrophyllite gaskets and mullite or MgO/spinel pressure media. We will test offline and then introduce online new designs with boron epoxy and/or boron nitride beam paths through these components. This will bring the absorption levels far lower and allow good access to a wide energy range (see Figure). We will optimize the designs so as not to affect the temperature capabilities or success rates of the experiments.

We will also work on careful dimensioning of the assemblies and specially made sample/standard capsules that will provide full access to the sample and standard with positioning reliably within the beam window. This is important to the rapid success of users who bring their projects to the beam line.

Success at the beam line depends on careful offline testing combined with significant online experience. In developing these assemblies, we will perform the offline tests at ASU, including furnace testing and post-run sectioning to check the sample positioning, before putting the assemblies to online use. Beam time is very precious and should be spent collecting data for the scientific objectives, so the primary testing should be done offline to the extent possible. We can also perform detailed absorption calculations and simulations of the expected x-ray data in order to ensure that the scientific objectives are attainable with our assemblies.

Other areas of online development will be in ultrasonics, deformation, and electrical conductivity experiments, and each of these objectives is described in the relevant sections.

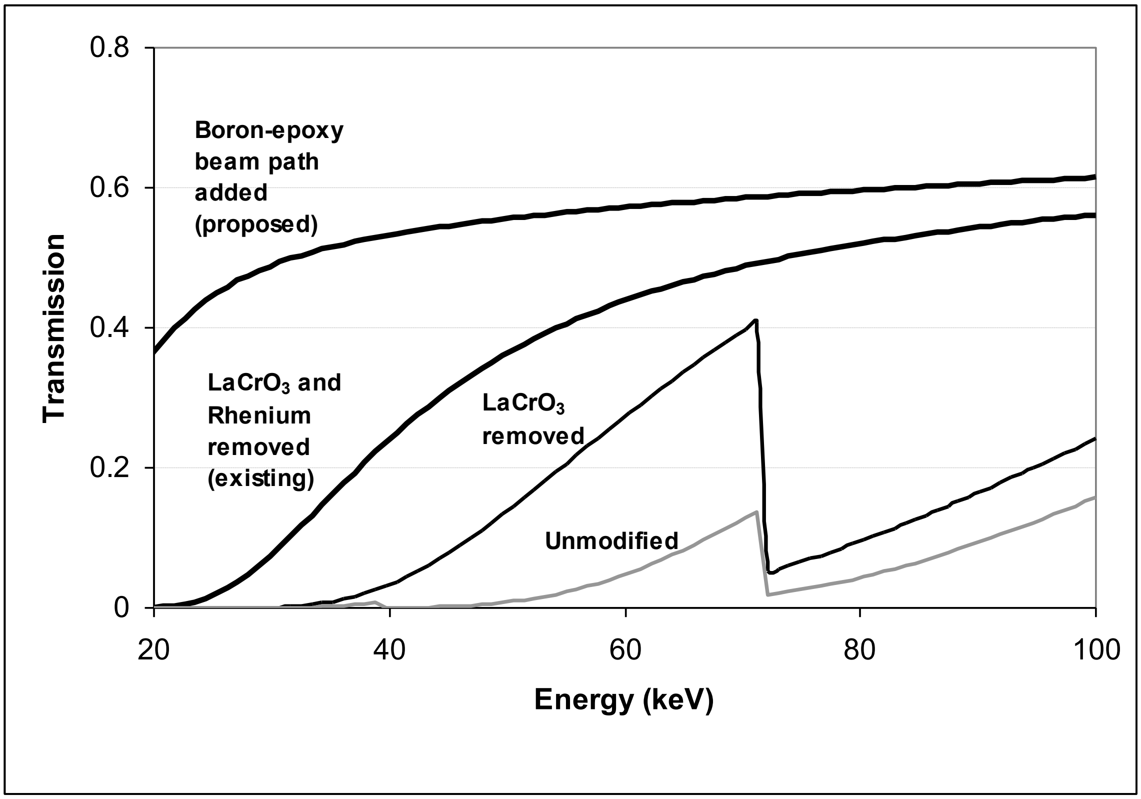


Figure: X-ray transmission curves for various versions of the 10/5 assembly, showing the current existing beam line assembly, and the proposed assembly with the addition of boron-epoxy beam paths through the octahedron and gasket.

Ultrasonics

The performance of ultrasonic measurements *in situ* is an objective of many groups, because of its application in comparing mineralogical models to seismic studies of the Earth. We will help to develop ultrasonic assemblies to provide to interested research groups. An ultrasonics setup at the beam line is currently targeted. Ultrasonic studies at the beam line are especially desirable because of the ability to measure pressure in-situ and to make a direct measurement of the sample length in real time, which enters into the calculation of the ultrasonic wave velocities. We intend to add ultrasonics assemblies to our list of standard assemblies available off the shelf. We will work with ultrasonics researchers to design the assemblies, test them offline, and then use them at the beam line. The ultrasonics equipment will stay at the beam line for outside users to access for their own research.

Deformation

Several forays into D-DIA research have been made by this project, including the use of mullite spheres or cubes as pressure media (Durham, Burnley) and the fabrication of provisional assemblies for D-DIA work (Holyoke). ASU does not have a DIA or D-DIA for testing, but will develop full D-DIA assemblies in consultation with D-DIA experts such as Pamela Burnley and with both the NSLS beam line and the GSECARS beamline, both of which have well-established D-DIA capabilities.

We have also been involved in helping with the new deformation 6-8 device at GSECARS with the Lesher group, and will assist with this project and develop new cell assemblies for that device as well.

Electrical Conductivity

We are currently working on a 14/8 *in-situ* electrical impedence cell assembly based on the designs of Scarlato et al. (2004). The small parts for the shielding and electrical leads are being made with the same techniques developed for the rest of the COMPRES assemblies, which will make it possible to manufacture the assemblies for widespread usage. This assembly will be available to the community after testing in electrical conductivity measurements.

Improved sample encapsulation

In the first phase of this project, we have often left it to the end users to encapsulate their samples, simply providing the assemblies with an empty space or "hole" where the sample and its capsule are intended to go. However, despite the complex and sample-specific nature of encapsulation in general, there are several standard ways to encapsule samples, and there are cost and other benefits in providing some of these to the community. For instance, precious metals are a significant cost and are not available in small pieces that would be needed for these experiments. It would be useful to develop a technique of precisely and cleanly cutting precious metal capsules into the precise lengths needed for multi-anvil experiments, allowing various compositions to be supplied in small, single-run quantities and eliminating the commitment that is usually needed to acquire precious metal tubing for sample encapsulation. We would work partly with Depths of the Earth, Inc. on this issue. Machined capsules, such as boron nitride, MgO, molybdenum, etc. are also a specialty item that some laboratories would prefer to have access to a supply of. And, single crystal capsules (MgO, forsterite, etc.) are of great and continuing interest to several groups using the COMPRES cells. Some of this has already been pursued by our project but we would like to make this a more standard and fully realized part of the project. Also, the discussion among different groups of encapsulation techniques for various types of samples will be a very useful one to have, and the actual procurement and development of such capsules will provide a concrete basis for such a discussion.

For the beam lines, the development of low-Z capsules, such as titanium, for in-situ hydrothermal experiments will be potentially very useful and will be pursued as part of this project.

Pressure Standards

It would be a significant service to the community to provide pressure standards to the community for high temperature fixed-point pressure calibrations. High-purity glasses and crystalline phases used for calibrations include SiO_2, Mg_2SiO_4, $MgSiO_3$, $CaGeO_3$, $ZnSiO_3$ and others. High accuracy can be obtained by using mixtures of the target phases, such as a mixture of coesite and stishovite for locating the coesite-stishovite transition near 9 GPa. This reduces the kinetic issues, acting as a self-contained reversal, and also minimizes the problem of large volume changes associated for example with the direct transformation of SiO_2 glass to stishovite, which can affect the pressure measurement. We will use the COMPRES project to obtain or synthesize high-purity materials that we can distribute to the community, thus providing a uniform set of pressure standards. Some of the samples can be made in large quantities in the belt apparatus at Diamond Innovations in Ohio (a division of Sandvik) and we have a collaborative relationship with this group, and will pursue the synthesis of large quantities of high-pressure phases of these pressure standards for community use.

Cell Assemblies for 5000 T press

In the event that COMPRES develops a 5000 Ton press for use by the high pressure community, we will assist that effort by developing larger cell assemblies for the press. Our largest cell assembly is currently 25 mm on the octahedral edge (octahedron/truncation sizes 25/15), but the methods used in that assembly are scaleable, and we can build even larger assemblies. On the large press at Ehime, in the synthesis of nanopolycrystalline diamond aggregates for anvils, octahedra/truncation sizes of up to 38/22 have been used to 15 GPa (Futoshi Idobe, pers. comm.), and we would develop similarly large assemblies using our injection-molded octahedron formulas.

Capital improvements

This project began in 2001 with the acquisition of an automated lathe for fabrication of cell assembly parts. Since then, the project has been entirely materials and supplies, and we have not requested capital items. However, the project can be improved by the acquisition of a modest amount of additional capital equipment that is directly related to the project, and we are requesting funding for this equipment as a Capital line item of $25K, in the first year only.

The machining of complex shapes such as spherical gaskets as used in the Spheres and Seats D-DIA design of Durham et al. (2008) fabricated by this project, prototype octahedra and pressure media such as test octahedra of mullite and other experimental ceramics, and other non-cylindrical parts would be greatly facilitated by having an automated and enclosed mini-mill such as the Roland MDX-40. We currently use more conventional large mills for this purpose, that do not have full x-y-z automation and thus cost a great deal of time and expense in labor in making such parts. Part of the Capital funding in the first year would be for this mini-mill.

The other capital improvement that would greatly assist this project is equipment that would allow automation and detailed tracking of voltage and amperage supplied to the furnace of the assembly, which was already mentioned above in the section on standard cell assemblies. We currently collect this information in a log book, but having it written to a computer in real time would allow us to discern more subtle unusual behavior in our assemblies. Quirks in the behavior (such as subtle resistance changes or changes in power-temperature relations) could then be traced to phase transitions in zirconia, faulting, localized melting, or other behavior by post-run sectioning and microprobe (backscattered electron) examination. This information would feed into design improvements in the assemblies.

Table 1: Multi-anvil assemblies developed by the COMPRES project

Assembly name	Peak pressure	Status	Proven temperature	Design
8/3	25 GPa	Available	2319 °C	Rhenium furnace
10/5	20 GPa	Available	2000 °C	Rhenium furnace
14/8 “G2”	13 GPa	Available	1200 °C	Graphite box furnace
14/8 step heater	15 GPa	Available	1400 °C	Graphite/$LaCrO_3$ step furnace
18/12	9 GPa	Available	1500 °C	Graphite box furnace
25/15	5 GPa	Available	1500 °C	Graphite box furnace
8/3 window assembly in-situ	25 GPa	Available	2200 °C	$LaCrO_3$ sleeve and rhenium furnace with windows
10/5 window assembly *in-situ*	20 GPa	Available	2200 °C	$LaCrO_3$ sleeve and rhenium furnace with windows
10/5 equatorial assembly *in-situ*	20 GPa	Available	1700 °C	TiB_2+BN straight furnace, MgO equatorial window, mullite octahedron.
14/8 “G2” *in-situ*	13 GPa	Available	1200 °C	Graphite box furnace, forsterite sleeve
14/8 equatorial assembly *in-situ*	15 GPa	Available	1500 °C	TiB_2+BN step furnace, MgO equatorial window, mullite octahedron

Table 2 – Laboratories and Investigators involved in the COMPRES Multi-Anvil Cell Development project through the continuing use of high-pressure COMPRES cell assemblies.

Institution	Location	Contact(s)/Advisors
Argonne National Laboratories	Lemont, IL	Tamas Varga
Arizona State University	Tempe, AZ	Kurt Leinenweber
Australian National University	Canberra, Australia	Robert Rapp
Bayerishches Geoinstitut	Bayreuth, Germany	Dan Frost
Brookhaven National Laboratories	Brookhaven, NY	Liping Wang
Brown University	Providence, RI	Stephen Parman, Geertje Ganskow
California Institute of Technology	Pasadena, CA	Jed Mosenfelder
Carnegie Institution of Washington, Geophysical Laboratory	Washington, D.C.	Valerie J. Hillgren, Li Zhang, Yingwei Fei
Case Western Reserve University	Cleveland, OH	James Van Orman, Katherine Crispin
China University of Geosciences	Wuhan, China	Zhenmin Jin
Chinese Academy of Sciences	Beijing, China	Changqing Jin
Columbia University, Lamont-Doherty Earth Observatory	Palisades, NY	David Walker, Wei Du
Delaware State University	Dover, DE	Gabriel Gwanmesia
Florida International University	Miami, FL	Jiuhua Chen, Helene Couvy
GFZ Helmholtz-Zentrum Potsdam	Hamburg, Germany	Monika Koch-Muller, Hans J. Mueller
Institute for Geowissenschaften, Universität Frankfurt	Frankfurt, Germany	Alan Woodland
IPGP	Paris, France	James Badro, Julien Seibert
Jilin University	Changchun, China	
Los Alamos National Laboratories	Los Alamos, NM	Jianzhong Zhang
Lawrence Livermore National Laboratories	Livermore, CA	Julien Siebert (moved to IPGP)
Massachusetts Institute of Technology	Cambridge, MA	William B. Durham, Nathaniel Dixon
NASA Johnson Space Center	Houston, TX	Kevin Righter, Lisa Danielson
National Cheng Kung University	Tainan, Taiwan	Jennifer Kung
Northern Illinois University	DeKalb, IL	Heather Watson
Stony Brook University	Stony Brook, NY	Baosheng Li, Hongbo Long, Li Li,

		Simon Hunt
Texas A&M University	College Station, TX	Caleb Holyoke
Toshiba Tungaloy America	Itaska, IL	Ron Pang
Yale University	New Haven, CT	Kazuhiko Otsuka, Justin Hustoft, George Amulele, Shun-ichiro Karato
Yanshan University	Qinhuangdao, China	Dongli Yu
University College London	London, England	Edward Bailey, Paul F. McMillan
University of Amsterdam	Amsterdam, The Netherlands	Wim van Westrenan
University of Arizona	Tucson, AZ	Kenneth Domanik
University of California at Davis	Davis, CA	Charles Lesher, Alisha Clark, Lara O'Dwyer Brown
University of Chicago,GSECARS	Argonne, IL	Yanbin Wang, Zhicheng Jing
University of Colorado	Boulder, CO	Joseph Smyth
University of Hawaii	Honolulu, HI	Murli Manghnani
University of Michigan	Ann Arbor, MI	Jie Li
University of Minnesota	Minneapolis, MN	Shenghua Mei, Tony Withers
University of New Mexico	Albuquerque, NM	Laura Burkemper, Carl Agee, Karen Hutchins, Stephen Elardo
University of Nevada at Las Vegas	Las Vegas, NV	Pamela Burnley
University of Western Ontario	London, Ontario, Canada	Richard A. Secco

Table 3: New multi-anvil assemblies in development by the COMPRES project in the near term

Assembly name	Target pressure	Proposed availability	Target temperature	Possible Design
8/3 higher T	25 GPa	Summer 2012	3000 °C	LaCrO3 furnace
10/5 higher T	20 GPa	Fall 2012	3000 °C	LaCrO3 furnace
10/4 in-situ XRD and ultrasonics	20 GPa	Spring 2012	2000 °C	Equatorial window, TiB2 + BN furnace
Improved x-ray access assemblies (8/3, 10/5, 14/8)		Spring 2012		Boron epoxy and/or BN x-ray beam paths added to existing assemblies
14/8 higher T	15 GPa	Spring 2012	3000 °C	LaCrO3 furnace
14/8 in-situ XRD and ultrasonics	15 GPa	Summer 2012	1400 °C	Graphite/$LaCrO_3$ step furnace
14/8 electrical conductivity	15 GPa	Fall 2011	1400 °C	Graphite/$LaCrO_3$ step furnace
18/12 lower power consumption assembly	25 GPa	Summer 2011	1800 °C	Porous mullite octahedron
25/15 lower power consumption assembly	20 GPa	Summer 2011	1800 °C	Porous mullite octahedron
6 mm D-DIA assembly	9 GPa	Fall 2011	1200 °C	Graphite furnace, with alumina deformation inserts

Publications resulting from use of the COMPRES cell assemblies

For the time period 2006 to July 2011, 31 publications and graduate student theses have resulted from COMPRES support of the multi-anvil cell assembly project. These are listed in the complete list of COMPRES publications submitted as a supplementary document to this proposal.

B.2.2 Applications of FIB/SEM CrossBeam Technology for High-pressure Research

COMPRES Infrastructure Development Proposal

PI: Yingwei Fei, Geophysical Laboratory, CIW

Scientific Goals and Technologic Advancements

Understanding the physical and chemical conditions and dynamic processes of the deep interior of the Earth has been a major goal of geoscience. High-pressure experiments provide essential data to interpret geophysical and geochemical observations. Very small sample sizes (micron to sub-micron diameters) are the characteristics of conventional ultrahigh pressure experiments that simulate conditions of deep Earth. Recent advances in synchrotron radiation techniques, particularly the improved capability of focusing x-ray beams to a few microns, have played important roles in studying the phase transitions and physical properties of materials under extreme high pressures and temperatures, including the discovery of post-perovskite at core-mantle boundary conditions [*Murakami et al*., 2004]. There has been significant development in probe techniques associated with powerful new-generation synchrotron radiation sources. These methods include x-ray diffraction, x-ray spectroscopy, inelastic scattering, radiography, and synchrotron infrared spectroscopy [*Hemley et al*., 2005]. These techniques have proven to be powerful in understanding the physical phenomena of materials under extreme conditions. What has been lagging behind in this revolutionary advance in high-pressure research is probing the chemistry of materials under extreme *P-T* conditions.

The use of laser-heated diamond anvil cells as reaction chambers for the study of chemical mass transfer is both necessary and nascent. Some recent studies have made progress in measuring major element partitioning between solid solutions by combining laser-heated diamond anvil cells with analytical transmission electron microscopic and other microanalytical methods [*Kobayashi et al*., 2005; *Murakami et al*., 2005]. These are technically challenging experiments with low success rates because it is extremely difficult to recover the very small samples involved and to prepare them for further analytical measurements. FIB techniques have revolutionized preparation of such specimens [e.g., *Heaney et al.,* 2001, *Benzerara et al*., 2005] and are providing unprecedented ability to quantitatively analyze recovered high-pressure samples [e.g., *Auzende et al*., 2008; *Ricolleau et al*., 2008, 2010; *Sakai et al*., 2009; *Frost et al*., 2010].

A state-of-the-art Carl Zeiss AURIGA CrossBeam FIB/SEM (focused ion beam/scanning electron microscope) system has recently been installed at the Geophysical Laboratory. The system is dedicated for high-pressure research and has been used for characterization of high-pressure samples, micro/nano-fabrication, and sample preparation for various analytical tools. The CrossBeam FIB/SEM Workstation integrates a FIB system and a field-emission scanning electron microscope (FE-SEM) in one powerful instrument. It equipped with an analytical Silicon Drift Detector (SDD) for chemical analysis, an Omniprobe 200.2 for automatic sample manipulating and in-situ lifting, and pneumatic retractable multi-modes STEM detector for detection of bright-field (BF) and dark-field (DF) signals generated by a thin specimen.

Using FIB technology, we can now extract thin film of the recovered samples precisely at the laser-heating spot (Fig. 1) and perform high-resolution SEM, STEM, and TEM analyses (e.g., Fig. 2). This will significantly advance our capability to probe the chemistry of the deep earth, leading to new scientific findings.

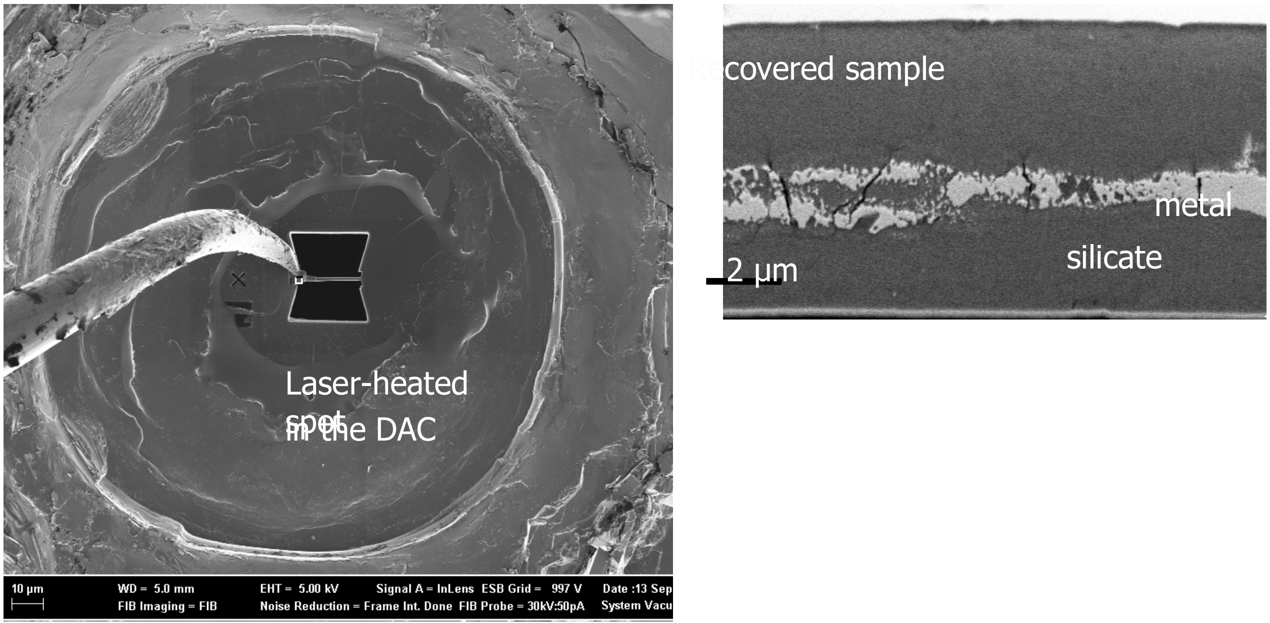


Figure 1. *SEM imaging of a recovered laser-heating sample in the diamond-anvil cell. The left-out lamella (8x18µm with a 100nm thickness) was prepared for composition and texture analyses.*

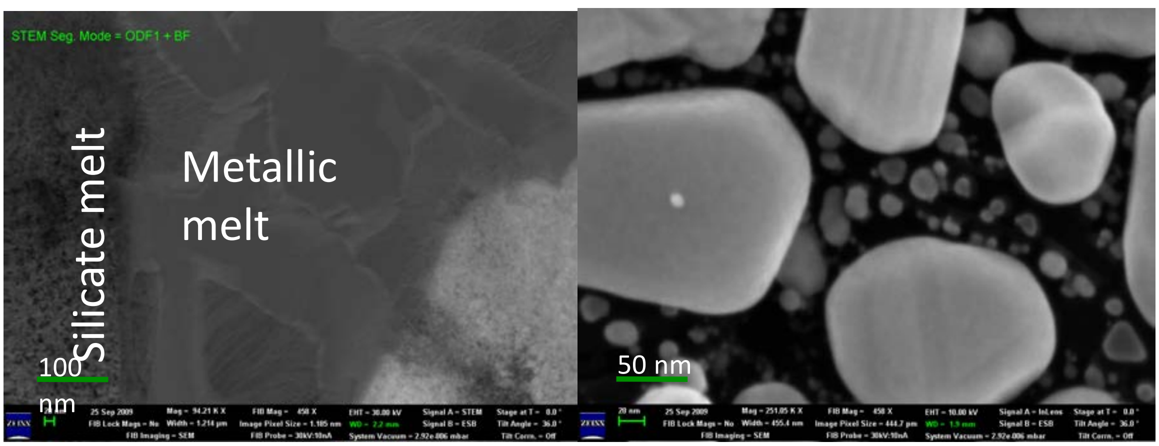


Figure 2. *a) STEM imaging of a lift-out TEM lamella. Orientational dark-field mixed with bright-field signal gives contrast between the silicate melt and the metallic melt. b) E-beam imaging of Au nano-particles.*

The AURIGA CrossBeam FIB/SEM Workstation is capable of providing 3D textural and shape information on mineral grains, inter-granular ground mass and cavities with tens of nm resolutions. This is the key to many geophysical problems, but we have essentially no knowledge for the bulk of the lower mantle and core. For instance, the seismic velocity and anisotropy in D" critically depends upon shape-preferred orientation (SPO) of individual minerals and fluid phases [*Lay et al*., 2004], but the only experiments conducted to date are on lattice preferred orientation (LPO). It is possible to calculate and predict LPO, while SPO relies solely on experimental observations. The dihedral angle at grain contacts and the fracture systems of rocks under shear are controlling factors for whether the rock will percolate or trap a fluid, and is the key to understand the formation of planetary cores, deep Earth volatile budgets, and geochemical cycles [e.g., *Minarik et al*., 1996; *Yoshino et al*., 2003]. Inter-diffusion coefficients among deep mantle phases dictate the time and length scale of chemical equilibrium and mass transport [*Holzapfel et al*., 2005]. With the greatly improved resolution, the FIB will enable all of these measurements on small multi-phase samples recovered in high *P-T* diamond cell experiments. Figure 3 shows 3D reconstruction of iron melt droplets in silicate melt.

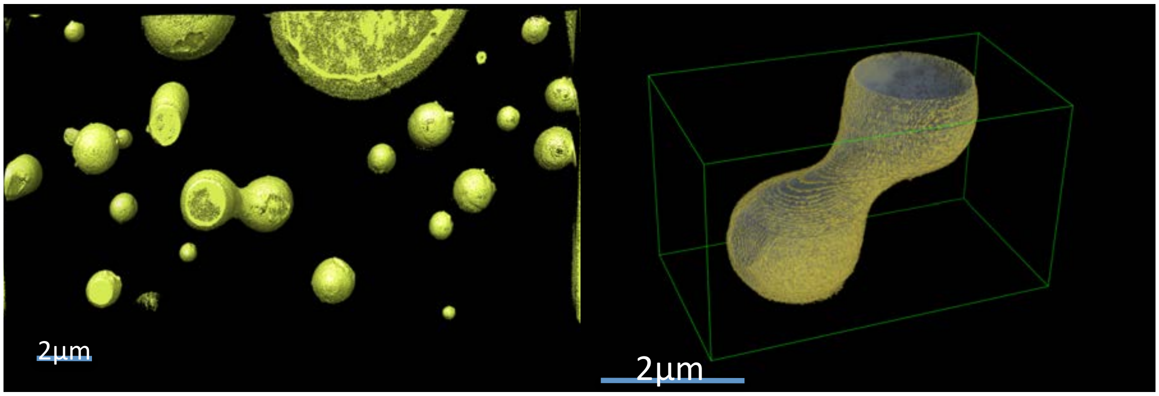


Figure 3. *Isosurface rendering of iron melt droplets in silicate melt. The images were reconstructed from 3D FIB serial sectioning and imaging from 92 slices with 20nm slice thickness.*

FIB technology opens new possibility for transport measurements in diamond anvil cells. It is possible now to develop a new process for preparing samples and electrical leads in a single step using FIB techniques. For example, FIB is used to deposit conducting Pt leads and attached these leads to the sample (Fig. 4). 4-probe resistivity measurements have been successfully performed to 20 GPa, and 2-probe measurements to 40 GPa [*Cuk et al*., 2008]. There are no technical limitations for the technique to work at much higher pressures because the FIB technique is capable of working with very small samples, down to the nanoscale.

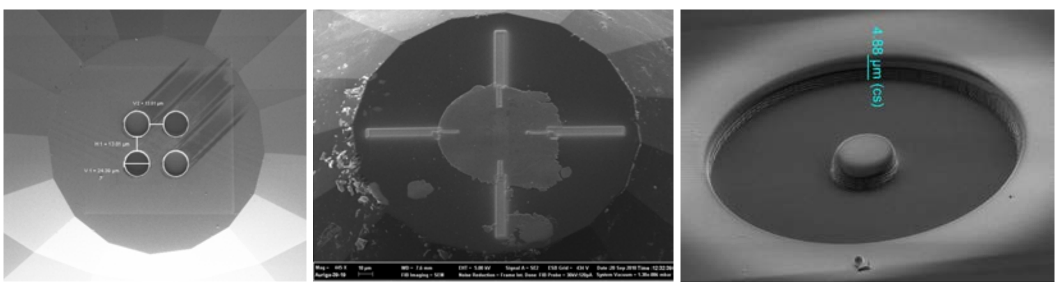


Figure 4. *Smart anvil and sample chamber design and Pt leads design for conductivity measurements.*

The FIB experimental environment allows very fine sample control and preparation. This functionality will be used to create a full sample arrangement for any conceivable transport, physical property, and chemical measurements at the micro-scale (10-30 μm or less) and eventually at the nano-scale, allowing a completely new suite of measurements under extreme conditions. For example, thermal transport, tunnelling, and even more sophisticated experiments may become possible in diamond anvil cells under quasi-hydrostatic conditions (*e.g.*, in He pressure media). New anvil designs will be possible based theoretical predictions. With FIB technology, we will create sample environments as a “microchip” on the diamond anvil, with contact pads extending to the outside connections on a larger scale (0.3-1 mm). These contacts can be interfaced easily with standard external equipment for measurements by standard “manual” techniques or using more complicated photolithography techniques. Such an approach will also work with designer anvils, which provide a convenient way to establish the electrical connection to the sample within the cell. The new procedure will enable us to explore transport properties of materials at high pressure on a routine basis and reach even higher static pressure with designed anvils.

The new AURIGA CrossBeam FIB/SEM Workstation is also capable of making STEM image of a left-out TEM lamella with 0.8nm resolution and ultra-high resolution e-beam images (Fig. 2a, b). With the analytical Silicon Drift Detector (SDD), we can also make element maps and provide quantitative chemical composition analysis with proper calibrations.

Enabling FIB Technology for High-Pressure Research

The use of FIB techniques has revolutionized the preparation of samples for micro- and nano-analysis in material sciences. The new generation of CrossBeam FIB/SEM system combines precision milling and ultra-high resolution and it is a versatile instrument. The principal emphasis of our FIB applications is to advance high-pressure research. We have identified several applications of FIB technology for deep earth study, including precise sample recovery from diamond cell and other high-pressure experiments for composition and texture analyses, 3D reconstruction of quenched high-pressure samples to study melt migration, and micro/nano-fabrication of smart anvil for transport property measurement and enabling new design of high-PT experiments. Many more new applications are possible with inputs and expertise of the COMPRES users.

Many COMPRES users may have access to FIB facility from their own institutions. The challenge for the high-pressure community is to access expertise to deal with delicate high-pressure samples and applications specific to high-pressure research. As a user of a materials science orientated FIB facility, it is often that the high-pressure user does not have enough time to develop FIB tools specific for high-pressure experiments or could not get a type of help needed to deal with high-pressure samples from traditionally trained FIB technician. The proposed infrastructural development program will provide training for COMPRES users to be FIB technology savvy for high-pressure research. The program will develop basic FIB tools specific for high-pressure research and the COMPRES users can take these skills to any FIB facility after training. We will also work with users to develop specific tools for their research. With COMPRES support for ½ time research associate, that person will be available to consult with users to develop FIB tools and help with use of the developed FIB tools for high-pressure research.

As part of community service and outreach, we will organize at least three hands-on training sessions per year (limited to 5-6 participants at each sessions for high-productivity). The COMPRES-supported research associate will work with individual after the training if needed. There are no charges for FIB time for these training sessions. In addition, we will consider training for individual COMPRES user upon request. The training sessions and user program will encourage broad collaborations and lead to new development of techniques which will be shared by the community.

During the last few years, FIB technology has played an increasingly important role in shaping high-pressure research, particularly in studying the deep Earth. The key aspect of this ID project is to power more COMPRES users to effectively use FIB technology solve deep earth problems. It will open doors for new research opportunities and greatly improve our scientific throughput. The involvement of COMPRES community will lead to novel and efficient uses of the technology and advance a number of scientific research fields.

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B.2.3 Mineral Physics on the World Wide Web – a comprehensive approach

COMPRES Infrastructure Development Proposal

Pamela Burnley and Sylvia-Monique Thomas, Department of Geoscience, University of Nevada, Las Vegas

Part I: Undergraduate Portal to Mineral Physics

We propose to oversee the development of a COMPRES outreach website (which will be a part of the main site) that will be designed to recruit undergraduates to graduate programs in Mineral Physics by providing information and networking opportunities.

The site would have several aspects:

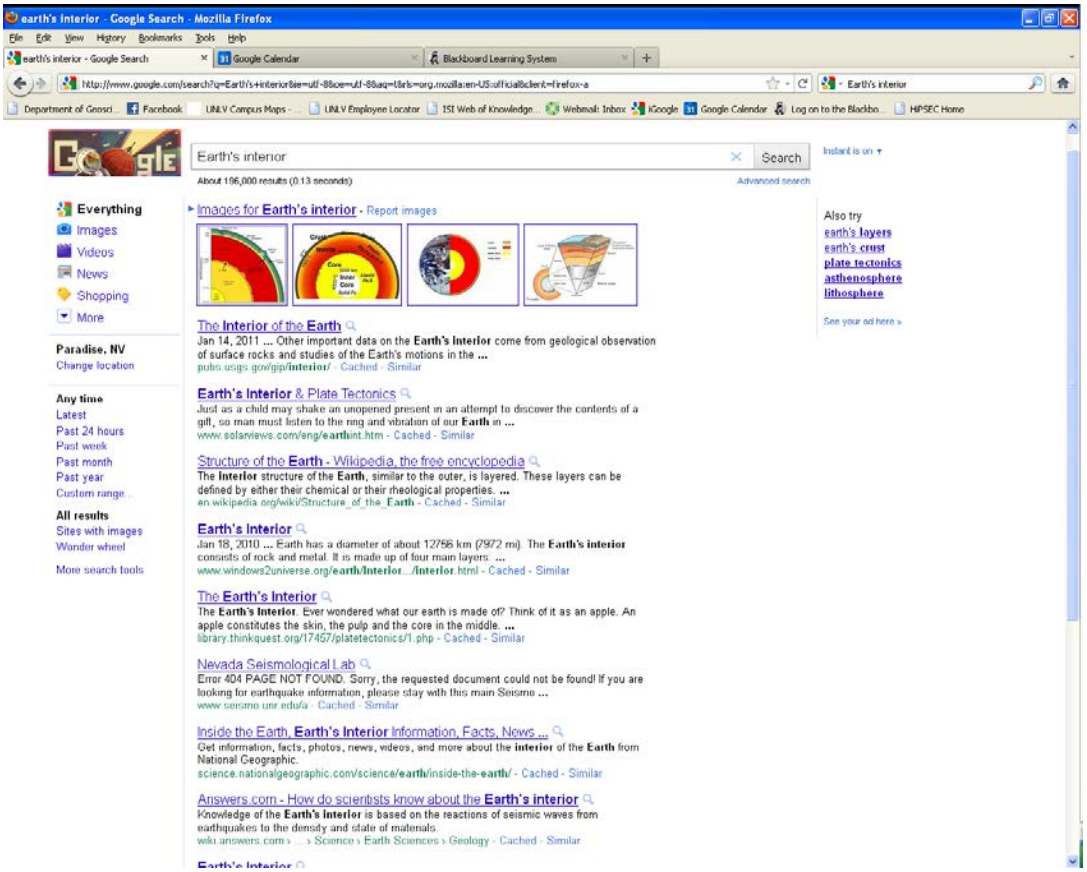
1. Interesting and appealing front page (lots of images)
2. Pages connected to the Distinguished Lecture Series (e.g. background information, where to get more information on topics in series)
3. Pages on how to pick a graduate program and connect with an advisor
4. A list of mineral physics faculty currently searching for graduate students
5. Pages explaining mineral physics topics. These pages would be "recycled" from the mineral physics distance education graduate course materials as well as from the Mineral Physics Wikipedia project.
6. Pages profiling mineral physics jobs including first person articles by beamline scientists, faculty, national lab researchers, etc. talking about what they do in their job and how they got there.
7. Introductory overview of COMPRES community facilities (with links to facilities pages).

Work plan

Burnley and Thomas would provide the content for the undergraduate portal and the COMPRES central office would assist with formatting and incorporating the content into the COMPRES web page.

Part II: Mineral Physics on Wikipedia: Placing mineral physics information in places where people are likely to find it

It would be difficult to overstate the importance of the internet in public outreach and education. Increasingly, both students and the general public rely on internet sources of information in all aspects of their life. However, finding a means of reaching people who are searching for the information that we want to share is non-trivial. A simple Google search on the term "Earth's Interior" yields 196,000 results:



The first Hollow Earth website appears on page 3. The first article related to Mineral Physics appears on page 4. Note that as with many searches, the link to Wikipedia occurs high on the first page. Wikipedia (according to itself) is *"a free, web-based, collaborative, multilingual encyclopedia project supported by the non-profit Wikimedia Foundation. Its 18 million articles (over 3.6 million in English) have been written collaboratively by volunteers around the world, and almost all of its articles can be edited by anyone with access to the site.[3] Wikipedia was launched in 2001 by Jimmy Wales and Larry Sanger[4] and has become the largest and most popular general reference work on the Internet,[2][5][6][7] ranking around seventh among all websites on Alexa and having 365 million readers."* In its description of itself, Wikipedia contributors are referred to as "non-experts", however, experts are not discouraged from contributing. Despite the fact that it is a general reference work, fairly esoteric topics, for example "Martensitic transformation", "Burgers vector", "Von Mises yield criterion " receive comprehensive and accurate coverage.

A quick search for COMPRES-related topics revealed spotty coverage. "Diamond Anvil Cell" has a good entry but there is almost no coverage of the multi-anvil apparatus. The diamond anvil cell entry does not mention geophysics as a possible use of the DAC. The entry for the Earth's interior covers seismology but does not mention mineral physics. There is an entry for "mineral physics", but we could not find any pages that link to that page, including the pages referenced in the mineral physics entry. Facilities such as GSECARS, CDAC and/or HPCAT have no entries. COMPRES is not mentioned anywhere in Wikipedia. In comparison, seismology has extensive coverage. Earthscope has a very lengthy entry (clearly provided by Earthscope). IRIS and UNAVCO both have entries. With 365 million readers, Wikipedia is not a bad place to put mineral physics information for people to find.

We propose to direct the COMPRES Wikipedia project. This project would encourage graduate students and postdocs to generate articles for Wikipedia and to link existing articles to their article so that their article can be more easily found. The articles would also be loaded on the COMPRES web site. COMPRES would facilitate this activity by providing editorial guidance and a small stipend for each completed work. Each article would be pre-approved and consist of a pre-defined minimum number of words, graphics and links placed on other pages. Stipends could be held centrally and the maximum number per year could be set by the ExComm. The director's role would be to:

1. Recruit students and postdocs to write the articles
2. Review and approve proposals for articles
3. Review article drafts and provide feedback on writing for the general public
4. Approve the distribution of the stipend upon completion of the article.

To kick off the project, Burnley and Thomas will generate new articles and modify or correct existing articles as well as assist with the editorial duties. In addition to building a permanent mineral physics presence on Wikipedia, the project would also help graduate students and postdocs improve their communication skills and encourage them to continue making contributions to education and outreach.

B.2.4 IXS-NRS: Unique capabilities for COMPRES

Inelastic x-ray scattering and nuclear resonant scattering under extreme conditions

2012-2017 COMPRES Infrastructure Development Project

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Introduction

High-resolution inelastic x-ray scattering (IXS) techniques provide the Earth and Planetary science community with unique opportunities for new and exciting results on the properties of materials at high pressure and temperature conditions. Our infrastructure development project is aimed at outreach to the COMPRES community on the capabilities and use of these techniques and at creating state of the art inelastic x-ray scattering techniques for characterizing the properties of materials under the high-PT conditions of planetary interiors, as well as geophysics and geochemistry of rocks, minerals, and meteorites. We intend to provide three different advanced techniques

1) Synchrotron Mössbauer Spectroscopy, **SMS** and imaging (Sector-3)
2) Nuclear Resonant Inelastic Scattering, **NRIXS**, and imaging (Sector -3)
3) Momentum resolved inelastic x-ray scattering, **IXS** (Sector 3 and Sector 30)

The demand for these technique steadily increased (Figure 1). The overall arrangement of these specific spectrometers is given Figures 2 and 3. The first two of these techniques provide a plethora of information regarding thermodynamics, elasticity and chemistry of iron bearing minerals. However, SMS and NRIXS are equally applicable to other isotopes like Kr, Eu, Sn, and Dy. Recently, there is an increased demand for Dy, Eu, and Sn related high-pressure work.
New prospects of implementing a mechanical chopper will increase the data throughput rates by two orders of magnitude, and development of Mössbauer microscopy will extend the scientific interest.
IXS technique, which measures phonon dispersion relations from single crystals, powders, glasses and liquids, is also maturing for geophysical purposes because of the advancements in x-ray focusing optics, and development of two advanced spectrometers at the APS: HERIX-3 and HERIX-30.
Both of these methods are in many ways ideally or even uniquely suited for addressing a number of important geophysical questions. While Nuclear Resonant Scattering, **NRS** provides information on electronic, vibrational, and elastic properties, such as the density of states and sound velocities, momentum-resolved IXS directly gives the dispersion relation of low-energy collective excitations to provide directional information on vibrational and elastic properties, such as the elastic tensor and sound velocities.

We propose to advance the applications of these techniques in the next 5-year phase by implementing

1. A new mechanical chopper to enhance the SMS experiments by a factor 100,
2. An upgraded HERIX instruments with increased number of analyzers, combined with in-situ x-ray diffraction and pressure-readout, and
3. A micron-resolution Mossbauer microscope facility for mineralogy.

Progress in the first 5 years: 2006-2011

During our first five-years of infrastructure development project, two full-time fulltime postdoctoral researchers, Dr. Michael Lerche and Dr. Hasan Yavas, finished their term and left the APS. Dr. Lerche is now the Associate Director for McClellan Nuclear Research Reactor at UC-Davis, CA, and Dr. Yavas was hired to build the PETRA-III Inelastic X-Ray Scattering beamline in DESY, Hamburg, Germany. We have hired a new postdoctoral fellow, a recent graduate from UIUC Geology Department, Dr. Lili Gao, who has finished her PhD thesis with COMPRES member, Prof. Jie Lie (now at Michigan University).

Also during this period, we have initiated high pressure experiments at the new IXS beam line (sector 30-ID) of the Advanced Photon Source and improved the experimental capabilities of the NRS and IXS beam line (sector 3-ID) to enhance its performance for high-pressure research. In particular, Sector 3–ID-C momentum-resolved IXS instrument has been converted for high-pressure measurements by adopting a new tandem focusing system, reducing the beam size to 18 micrometers with very high efficiency. Similarly, focusing efficiency for the NRS experiments in the 3-ID-B station has been improved by implementing a short-focus bimorph mirror. Immediately following these developments, the high-pressure research activity at HERIX- 3 and 30 picked up, as evidenced by the increase in the number of proposals submitted (see Figure 1), and hence we are offering both of these instruments as infrastructure for high-pressure research.

We have engaged in outreach activities, e.g., three presentations at the COMPRES annual meeting in June 2010 and international conferences to broadly disseminate information on applications of NRS and IXS to understand Earth materials. In particular, we accomplished the following:

1. Hired three fulltime postdoctoral researchers between 2006-2011.
2. Provided support for generating of user proposals for sectors 3-ID and 30-ID by COMPRES members,
3. Created new high-pressure capabilities at for IXS at 3-ID-C by implementing tandem focusing of toroidal and K-B mirrors,
4. Developed a new DAC set-up for sound anisotropy measurements, and an adaptor for panoramic DAC gas-loading,
5. A new panoramic membrane cell has been designed, ordered, and received.
6. Mössbauer Spectroscopy laboratory has been activated to serve high-pressure user community.
7. Organized workshops and CONUSS schools for the COMPRES community.

Up through June 2011, Dr. Lili Gao was involved with helping users to install CONUSS and PHOENIX programs, including troubleshooting, helping a number of graduate students from University of Texas, University of Michigan, MIT, Caltech, and Princeton. She maintains and improves two high-pressure research programs: PRESSURE SCALE and FTIEOS. She has designed, built and integrated beam absorbers into station 3-ID-B as part of the beamline component last year; ans she is building another beam absorber for stations 3-ID-C and D. She also assisted Dr. Ercan Alp to upgrade the Mössbauer Spectroscopy laboratory, by separating the two existing systems so they now function independently, designing and implementing the new setup dedciated to DAC's, setting up computer control of the new Mössbauer setup. Dr. Wenli Bi will assume the position that Dr. Lili Gao vacated (for personal family reasons) starting in October 2011.

During the last year, we have explored the possibility of reducing the size of the x-ray beam at the momentum-resolved IXS station in sector 3-ID. Drs. Yavas and Ahmet Alatas contributions were crucial in this effort. We were able to obtain a 17x20 μm^2 spot size (prior 150x150 μm^2) with the highest spectral flux density of any IXS beam line in the world, as shown in Figure 4. Thus high-pressure studies using momentum-resolved IXS at 3-ID became feasible as shown in Figure 5. Presence of COMPRES funded post-doctoral researchers is essential for the increased access.

Proposed work for 2012-2017 Period

We will continue to offer special capabilities for SMS, NRIXS, IXS (HERIX-3) and x-ray diffraction, XRD, in 3-ID-B and 3-ID-C stations, combined with laser heating or external heating. We are adding HERIX-30 spectrometer as a new tool available for the COMPRES community, located at Sector 30. We will provide data analysis help with CONUSS, PHOENIX, FIT-2D, and FTIEOS.

In order to continue to engage the COMPRES community, we need to improve our facilities. In particular, with the complexity of the systematic studies, requiring larger pressure, temperature and composition range, we need to cut down the experimental time. For SMS, this would be possible with the introduction of a mechanical chopper. The idea here is to use a mechanical shutter to block the nonresonant portion of the incident beam, which is 10^7-10^9 times stronger than the nuclear resonant part of the beam. The feasibility of this approach has been determined (T.S. Toellner, et al, J. Synchrotron Rad. vol. 18, 100 (2011)).

In the extension 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 their high-resolution IXS experiments. In this context, we make CONUSS package available for nuclear forward scattering (NFS) data evaluation, PHOENIX package for nuclear resonant inelastic scattering measurements (NRIXS), as well as FitEoS for equation of state.

We intend to organize a workshop in May 2011, in connection with the APS User Meeting, to introduce the high-resolution IXS and its applications for studying planetary interiors with emphasis on attracting graduate students and young scientists. 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.

For NRIXS experiments, our current laser system needed replacement with a fiber laser. While we have acquired a new one with the APS funds, it turns out that we need a second one for two-sided heating. This is due to special unpolarized nature of the fiber laser. Furthermore, for high-pressure melting experiments, we need faster temperature readout than what the current spectrometer is capable of. In collaboration with Jennifer Jackson (Caltech) we have designed a new system, which is ready to be installed. This system relies of blackbody radiation profile to measure limited number of wavelengths, and fitting the data to a temperature, all the necessary optics and data acquisition tools are in place. The system will be installed in January 2011. We expect to push the temperature readout time to a few Hz.

We are also installing web-based cameras for remote-access and classroom access. This will provide COMPRES PI's to observe the experiments from their labs, participate the experiments in a more meaningful way, as well as demonstrate to the students who are not taking part in the experiment itself for that particular run period. If successful, we may plan, for limited experiments, remote-access operations for data collection, and on-line data evaluation. This should further expand the experimental domain in the COMPRES community.

We will improve the isomer shift measurements by developing a new method, which includes shifting the reference sample resonance line position by employing a constant-velocity drive. We are the only beamline in the world, offering all of these capabilities as part of our general user program.

One major limitation so far for SMS measurements has been the speed with which the data is collected. In many cases, samples with dilute iron concentrations in very thin samples, which are typical for high-pressure work, requires a few hours of data collection time. This can be reduced to a few minutes by employing a mechanical chopper. The proof-of-principle experiment shows that this is indeed the case, and data collection rates can be increased by a factor of 100. This will require the development of a mechanical shutter, schematically depicted in Figure 6, and the results are demonstrated in Figure 7.

Recently, in 2010, we have introduced new high-pressure set-up in Sector 3-ID-C station for momentum-resolved IXS measurements. The COMPRES members now routinely apply the HERIX-3 spectrometer at Sector 3, and HERIX-30 spectrometer at Sector 30-ID-C for momentum-resolved IXS measurements. Improving the in-situ XRD capability, in combination with the IXS spectrometer in 3-ID-C station is a priority. The added diffraction capability will provide us with structural confirmation as well as with an equation-of-state.

Additionally, the MERIX instrument at 30-ID-B station is receiving requests for resonant inelastic x-ray scattering under high pressure. At least, three such proposals have been submitted. It will be fair to recognize that the COMPRES activities related to inelastic x-ray scattering is growing in its reach and depth. Experiments above 1 Mbar hove become routine. More and more, we see "difficult" samples with very low iron concentrations are being measured. Polycrystalline samples to measure longitudinal sound velocities are being brought in, with good results, as seen in Figure 5.

Finally, we now offer the use of laboratory based Mössbauer spectrometer, suitable for high-pressure work, and data analysis. This facility is particularly very useful in developing strategies for data analysis obtained at the beamline by characterizing the samples ahead of time, as well as providing starting parameters for the data analysis of the time domain SMS. Sample purity, and valence state is an issue that beset every experiment, and the availability of this laboratory year round is an asset for COMPRES community. In addition to iron, we will continue to offer capabilities in Kr, Eu, Sn, and Dy (see Table 3 for all Mossbauer transitions observed at a synchrotron to date).

On the instrumentation side, we will improve the HERIX spectrometers performance by a set of measures, including change of undulator period and incident energy, by increasing the number of analyzers and analyzer configuration (see Figure 8) and by adapting new strip detectors as they become available.

General User Proposals (GUP) to APS

In the time period of 2008-2010, we have seen an increase in the number of proposals submitted, as well as a significant increase in beam time allocations. 12 independent research groups from 10 COMPRES member institutions submitted a total of 76 proposals to 3-ID and 30 proposals to 30-ID.

Table 1. The general user proposal entry summary for high-pressure related activities in dedicated inelastic x-ray scattering beamlines of 3-ID and 30-ID.

Years	3-ID Allocated	3-ID Submitted	30-ID Allocated	30-ID Submitted
2008	9	16	1	4
2009	11	20	3	5
2010	12	18	5	10
2011(*)	15	22	7	11
Total	**47**	**76**	**16**	**30**

(*) Projected based on first trimester of 2011

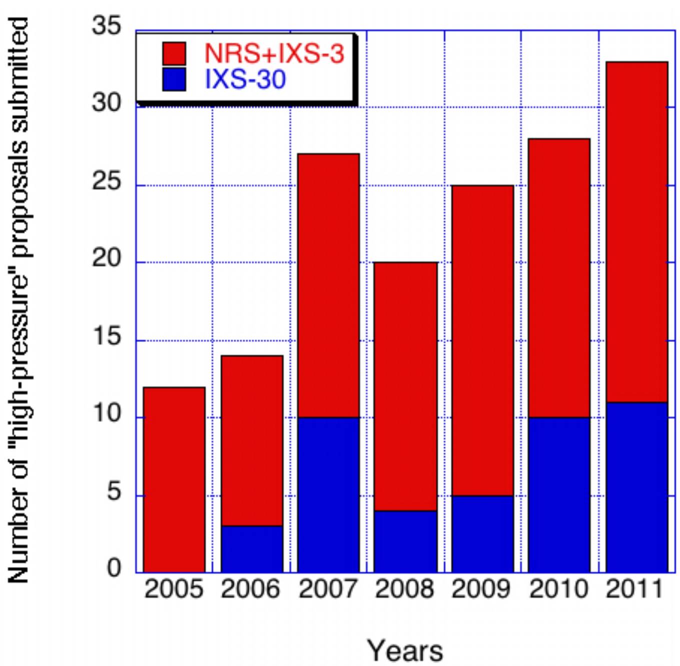


Figure 1. The historical development of high-pressure related research proposals submitted through the APS General User Proposal system. The number of the 2011 proposals is based on the projection of the first cycle.

Of these proposals 47 (= 62 %) and 16 (= 53 %) were granted beam time at Sector 3-ID and 30-ID, respectively (see Figure 1). These percentages are above the average acceptance ratio, which is below 30. This demonstrates a relatively higher success rate for COMPRES proposals. COMPRES funded post-doctoral researchers play a pivotal role for this enhanced ratio of acceptance. In almost all proposals granted beam time, students at graduate or undergraduate levels participated actively. The high success rate of proposals by COMPRES members demonstrates that Dr. Hasan Yavas and now Dr. Lili Gao worked well with many of the PIs to develop effective proposals that were very competitive for beam time. Further details about proposals that were submitted but not allocated beam time are considered confidential information by the APS and cannot be distributed in cross-institutional reports.

Table 2: COMPRES affiliated and other universities and research organizations that were allocated beamtime in 2009-2010 period (or will be in 2011-1 period), many of them repeatedly.

Affiliation	PI	Student
Caltech	J.Jackson, B. Fultz	Yes
Princeton	T. Duffy	Yes
MIT	S. Shim	Yes
U. Texas-Austin	J.F. Lin	Yes
ORNL	O. Delaire	Yes
Stanford	W. Mao	Yes
Carnegie Institute of Washington	Y. Ding, D. Mao V. Struzhkin	Yes
Washington University St. Louis	J. Schilling	Yes
University of Chicago	N. Dauphas	Yes
U. of Hawaii	M. Mangahni	Yes
UNLV	A. Cornelius	Yes
UIUC	J. Bass	Yes
Michigan	J. Lie	Yes
NRC	D. Klug	
U. Saskatchewan	J. Tse	
LLNL	D. Farber	
ISU-Ames	R. McQueeney	Yes
WUSL	J. Schilling	Yes

Publications (2006-2011)

For the time period 2006 to July 2011, 30 publications and graduate student theses have resulted from COMPRES support of inelastic X-ray scattering at Sector 3 of the APS. These are listed in the complete list of COMPRES publications submitted as a supplementary document to this proposal.

Workshops:

As requested by the COMPRES Executive Committee we removed any requests of participant support for workshops on IXS and NRS. It is our understanding that COMPRES would seriously consider such requests at a later time. These workshops would be widely advertised and emphasize outreach to students and young scientists. We expect to get approximately 30 participants per workshop and participant support would be used mainly to cover registration fees (including some meals, costs of space)

Table 3. Mössbauer transitions observed by synchrotron radiation given with their transition energies, half-lives, and first reported observations. The required optics is only available for highlighted isotopes at Sector 3.

Isotope	Energy (keV)	Half-life (ns)	First published reference
^{181}Ta	6.2155	9800	A. I. Chumakov, et al, Phys. Rev. Lett. **75** (1995) 16384
^{169}Tm	8.4013	4	W. Sturhahn, et al, Europhys. Lett. **14** (1991) 821
^{83}Kr	9.4035	147	D.E. Johnson, et al, Phys. Rev. B **51** (1995) 7909
^{57}Fe	14.4125	97.8	E. Gerdau, et al, Phys.Rev.Lett. **54** (1985) 835
^{151}Eu	21.5414	9.7	O. Leupold, et al, Europhys. Lett. **35** (1996) 671
^{149}Sm	22.496	7.1	A. Barla, et al, Phys. Rev. Lett. **92** (2004) 066401
^{119}Sn	23.8794	17.8	E. E. Alp, et al, Phys. Rev. Lett. **70** (1993) 3351
^{161}Dy	25.6514	28.2	A. I. Chumakov, et al, Phys. Rev. B **63** (2001) 172301
^{201}Hg	26.2738	0.63	D. Ishikawa, et al, Phys. Rev. B **72** (2005) 140301
^{129}I	27.770	16.8	unpublished
^{40}K	29.834	4.25	M.Seto, et al, Hyperfine Interact. **141** (2002) 99
^{125}Te	35.460	1.48	H.C.Wille, et al, Europhys. Lett. **91** (2010) 62001
^{121}Sb	37.129	4.53	H.C. Wille, et al, Europhys. Lett. **74** (2006) 170
^{61}Ni	67.419	5.1	T. Roth, et al, Phys. Rev. B **71** (2005) 140401
^{73}Ge	68.752	1.86	M.Seto, et al, Phys. Rev. Lett. **102** (2009) 217602

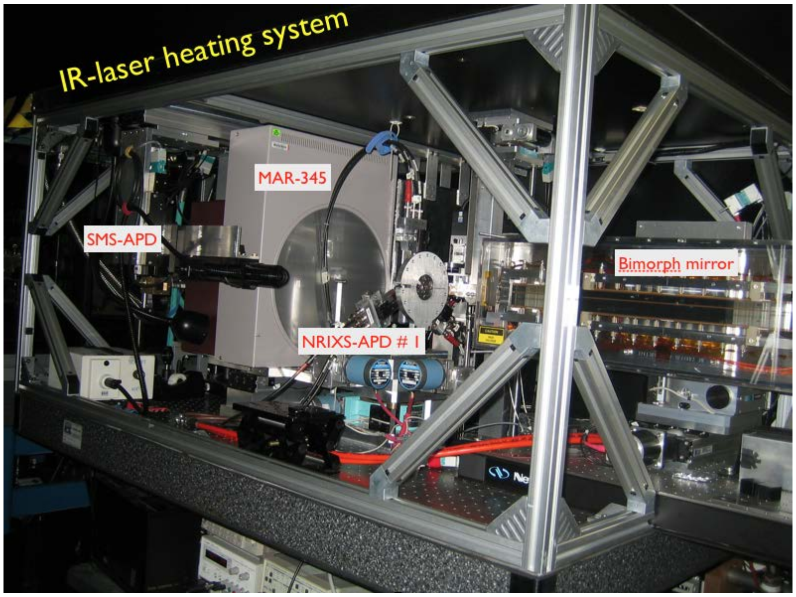


Figure 2. The nuclear resonance station dedicated to high-pressure research, offering simultaneous studies with synchrotron Mössbauer spectroscopy, SMS, nuclear resonant inelastic x-ray scattering, NRIXS, x-ray diffraction, XRD, and imaging with newly developed Mössbauer microscope. The laser heating system is being upgraded to fiber laser, and fast temperature readout system.

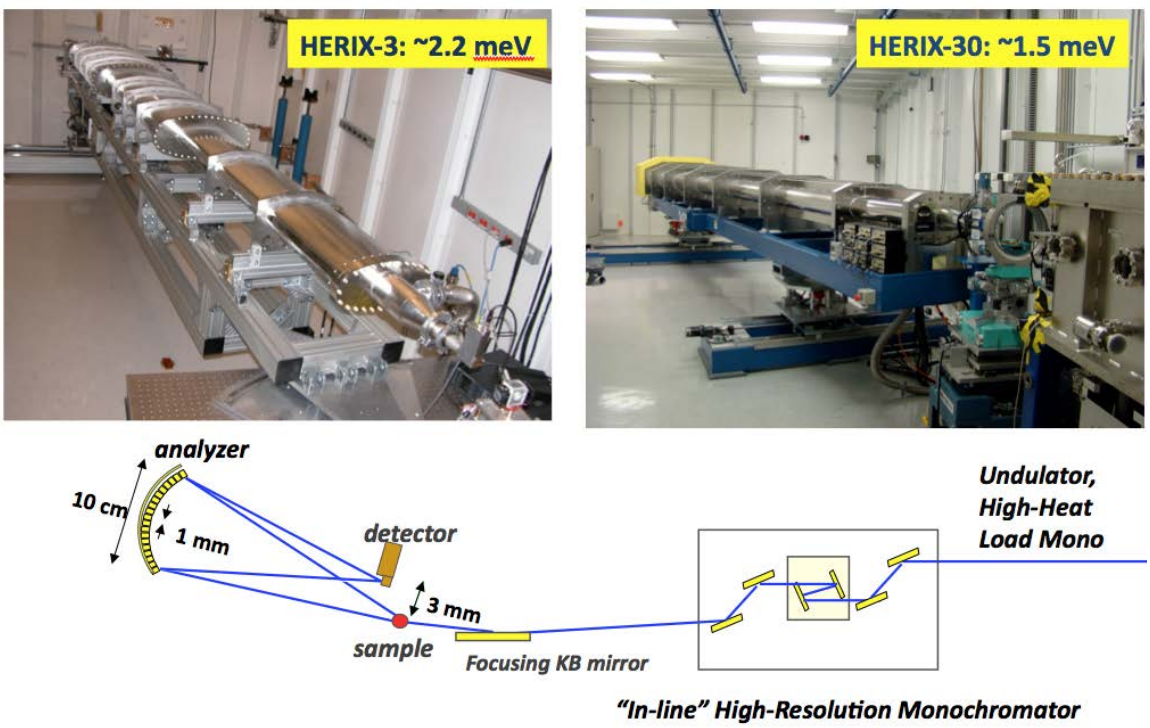


Figure3. The momentum-resolved inelastic x-ray spectrometers, HERIX-3 and HERIX-30. Both of these spectrometers have integrated microfocusing systems, suitable for high-pressure DAC work.

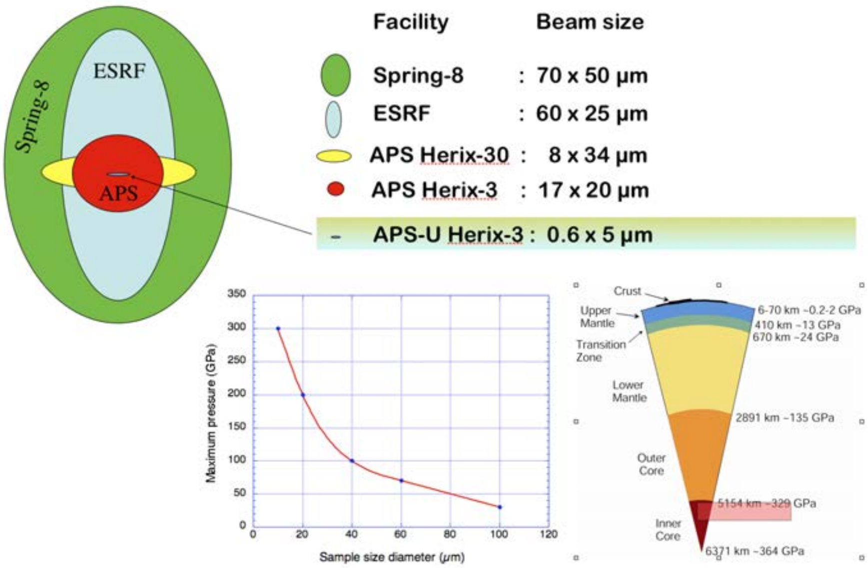


Figure 4. Comparison of full-focus beams size for all momentum-resolved IXS instruments in the world, which is a total of 4. Both instruments at the APS represent a unique opportunity for the COMPRES community, especially for experiments above 1 Mbar.

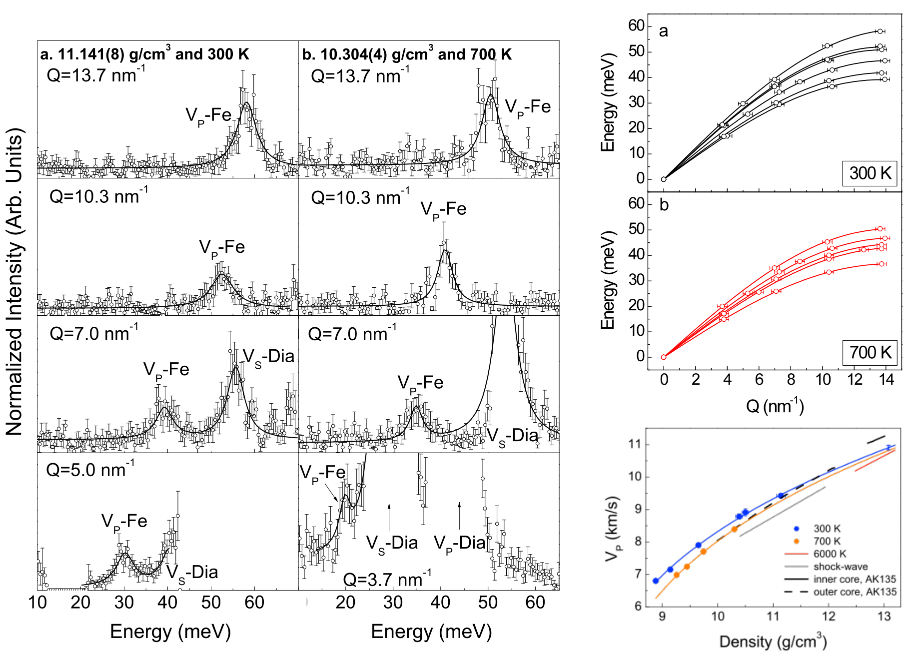


Figure 5. The new capability at Sector 3-ID-C IXS instrument is used to measure the phonon dispersion curves of hcp-Fe at high P-T. On the right, (a), ϱ = 11.141 g/cm3 at 300 K, corresponding to 105 GPa;
(b), ϱ = 10.304 g/cm3 at 700 K, corresponding to 67 GPa. Blue and orange lines: modeled V_P at 300 K and 700 K from the IXS results, respectively. On the left, top, 4-analyzer results for phonon frequencies at different momentum-transfer points inside the Brillouin Zone obtained at two different temperatures, and bottom, V_P –ϱ relation of hcp-Fe in Earth's core. (Z. Mao, submitted to Nature)

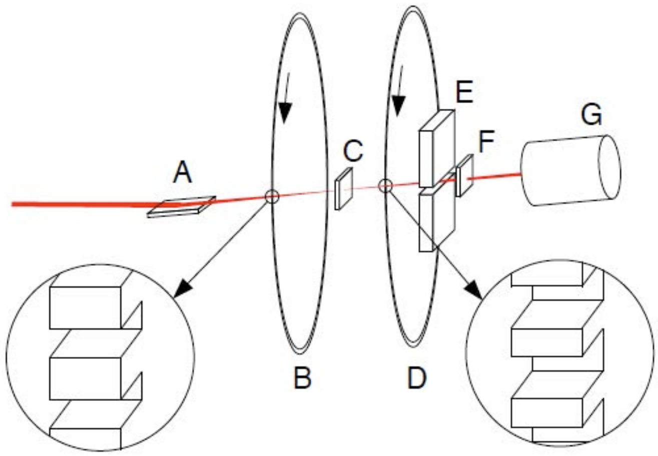


Fig. 6 Proposed setup for performing SMS using high-speed shutters. Focussing system (A), anti-shutter (B), sample or near-single-line resonant absorber (C), shutter (D), slits (E), sample or near-single-line resonant absorber (F), detector (G).

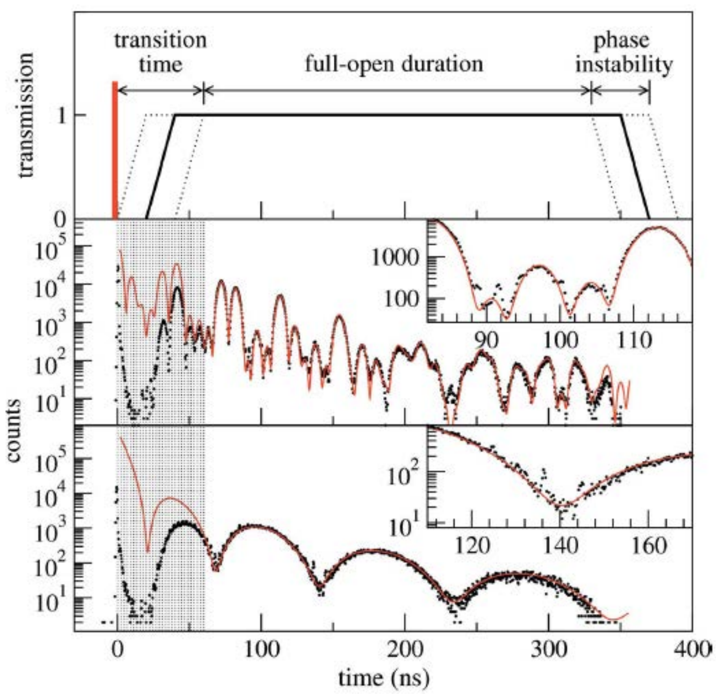


Fig. 7. SMS measurements in the time domain using a fast shutter. The top panel shows the approximate transmission window of the shutter on a time scale; zero time corresponds to the X-ray excitation pulse. The middle panel shows the time spectrum of nuclear resonant decay in the forward direction of a 3 mm-thick _-Fe foil enriched with 95% 57Fe. The bottom panel shows the same for a 12 mm-thick stainless steel foil enriched with 95% 57Fe. Solid lines are simulations assuming the shutter opens immediately and help to demonstrate that the transmission window is fully open 60–330 ns after nuclear excitation. Insets are magnified regions that show contamination from spurious X-ray pulses (C.7).

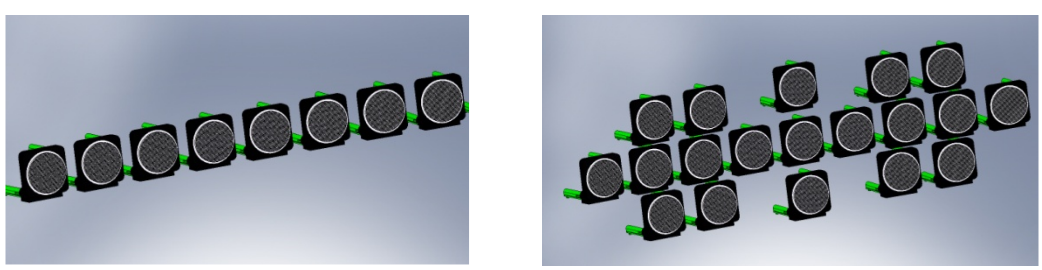


Figure 8. Improved analyzer configurations like the ones shown above will keep HERIX spectrometers competitive for the years to come. Such innovative ideas are motivated for both single crystal elastic tensor measurements as well as for polycrystalline compressional sound velocity measurements.

B.2.5 Post-doctoral position for the gas-loading system at APS

COMPRES Infrastructure Development Project
Mark Rivers
Sergey Tkachev
University of Chicago

The COMPES Infrastructure Development Committee funded the capital equipment costs (~$85,000) of a gas-loading system at the APS. GSECARS contributed the design and construction effort to build the system. The system began operation in February 2008 and has been running with minimal downtime since then. The system works extremely well, with the only significant problems being some failures of the commercial compressor. We have in-house technical support (Guy Macha) to repair such problems, and the mean time to repair has typically been 1 day.

The COMPRES system at the APS is available for use by any member of the COMPRES community, regardless of whether they are performing experiments at GSECARS, at another APS sector, at another synchrotron, or in their home laboratory. The problem for some members of the COMPRES community is that they need to load cells, but cannot afford the time or money to travel to APS.

GSECARS has provided the support (training and supervision) for any users who come to GSECARS to use the system. This is a substantial time commitment for our staff, but one which we can manage with our existing staffing level. We do not, however, have the staff to be able to handle a “mail-in” service to load cells for users; we rely on users to do most of the work once they have been trained. We also do not have the staff to provide assistance to users of other beamlines at the APS, such as the high-pressures users from sectors 3 (inelastic), 4 (magnetism), 16 (HP-CAT), or 32 (imaging).

We propose to continue the COMPRES support for 50% of a post-doc to reside at the APS. This person is responsible for loading cells that are sent to the APS by users who do not travel here, and for assisting users of other APS beamlines with loading their cells. The other part of this person’s salary and responsibilities will be covered by GSECARS.

Dr. Sergey Tkachev began in this post-doc position in the second week of June, 2010. He has performed an excellent job, providing both mail-in service for the COMPRES community not running at the APS, and hands-on assistance for users who come to the APS to load cells when running on any of the beamlines here.

Figure 1. Dr. Sergey Tkachev with the COMPRES/GSECARS gas loading system

The following table summarizes the mail-in service in the current APS run cycle, from February 1, 2011 to April 14, 2011.

University	Name	Number of DACs loaded
UCLA	Miao Xie	3
UNLV	Barbara Lavian & John Howard	5
Ohio State University	Daniel Reaman	4
LBNL	Simon Clark	2
Caltech	June Wicks	1
TOTAL		**15**

A total of 15 diamond-anvil cells have been loaded in this 2.5 month period for 5 user groups with the COMPRES supported mail-in service.

In addition to this mail-in service, there have been 165 users who have loaded cells in this same 2.5 month period. 75 of these users were performing experiments at GSECARS, while the remaining 90 were performing experiments at other APS sectors. 127 of these users were directly assisted by Sergey in loading their cells. Thus on average he is assisting more than 2 users with gas loading every working day. Sergey spends more than 50% of his time assisting such users and doing the mail-in service cells.

The following is a description of the mail-in service, as presented at 2010 COMPRES Annual Meeting:

- Mail-in service is available only for diamond anvil cells (DACs) that are being used for experiments at locations other than the APS.
    - Users who wish to load cells for experiments at the APS, including GSECARS, HPCAT and other APS beamlines, are expected to load the cells themselves.
    - Training and assistance will be provided by the staff from the beamline where the experiment will be conducted.
- Potential users must contact Sergey (tkachev@cars.uchicago.edu, 630-252-0430) to discuss the technical and scheduling requirements prior to sending the cells to the APS.
    - At this time we are providing mail-in service only for Ne and He gases in the standard symmetrical (Princeton) DAC
    - For all other type of DACs and gases the available options should be discussed directly with Sergey.
- Cells must be shipped with appropriate packaging that can also be used to return the cells.
    - A prepaid shipping label must be included for returning the cells to you.
    - Cells containing any hazardous materials must be not be shipped directly to GSECARS, but rather to the hazardous materials receiving building at Argonne.
    - Contact Sergey or Nancy Lazarz (lazarz@cars.aps.anl.gov) for details.
- We will make every effort to load the cells with the requested gas at the desired pressure.
    - However, COMPRES and GSECARS cannot guarantee that the cell will be successfully loaded.
    - The loading process also entails some risk of damaging the cell or diamonds. Users accept this risk, and agree not to hold COMPRES or GSECARS, or any of their staff, liable for any damage that may occur.
- Mail-in service is available only to staff and visitors at COMPRES member institutions in the United States, with shipping to and from US locations only.

Publications

For the time period 2008 to July 2011, 25 publications and graduate student theses have resulted from COMPRES support to build the gas-loading device at the APS under an Infrastructure Development project, and the follow-up post-doctoral support. These are listed in the complete COMPRES publication list submitted as a supplementary document to this proposal.

B.2.6 High Pressure-High Temperature Single-Crystal Diffraction Program at the ALS

Oliver Tschauner, University of Nevada at Las Vegas

The single-crystal diffraction project at the Advanced Light Source is one which has been supported by COMPRES at a very modest level, but will likely yield substantial returns and make the COMPRES-supported beamline 12.2.2. attractive to a broader range of users. A group led by Tschauner is installing the single-crystal diffraction capabilities that have been installed at GSECARS by P. Dera. COMPRES has paid a portion of the travel costs for Tschauner, Dera, and Barbara Lavina to install and commission the necessary software and related beamline modifications. This project builds on the strengths in single-crystal diffraction at beamlines 12.2.2 and 12.3.2 to bring a new capability to the West-coast synchrotron community, and should reap significant scientific dividends in the coming years.

Rock densities extracted from seismic data are accurate to within 1-2% (on a length scale of 200-300 km). This level of accuracy has allowed for observing heterogeneities in the lower mantle of the Earth. However, it remains unclear whether such heterogeneities are thermal or chemical in nature, or both. The PVT equations of state (EOS) of rock-forming minerals in the lower mantle should allow thermal and chemical heterogeneities to be distinguished from one another. At least, this is true if the EOS's are sufficiently precise. However, compilations of EOS data from several studies show that volumetric measurements by X-ray diffraction on lower mantle minerals have uncertainties of 5-10%. Volume variations at that level correspond to temperature differences of several hundred K and render the data of only qualitative use at best for geodynamical models.

This projects aims to address a major problem of experiments on the EOS of mantle minerals: The presently used powder diffraction methods on polycrystalline samples have intrinsic limitations in establishing significantly smaller errors because of an unavoidable lack of truly hydrostatic conditions. Most mantle minerals possess high elastic moduli even at mantle temperatures. Consequently local stresses in polycrystalline samples of these minerals deviate markedly from hydrostatic pressure. Any powder-diffraction analysis of such a sample reflects this stress variation, which results in diminished precision in the EOS.

We address this fundamental experimental issue in two ways: a) characterize the strain of each crystallite in a polycrystalline sample; b) examine single crystal samples in hydrostatic pressure transmitting media. Both pathways are built on single crystal- rather than powder diffraction. Single crystal diffraction has several fundamental advantages over powder diffraction:

- Hydrostatic compression can be established for single crystals but not for polycrystalline samples, as mentioned above.
- Higher accuracy: In single crystal diffraction distances and angles between lattice planes are quantified, but in powder diffraction only distances are measured. Proper assessment of lattice strain and of the accuracy of cell refinements requires measurement of angles.
- Higher precision from better statistics: Single crystal diffraction with diamond cells and synchrotron radiation provides up to several hundred reflections, while powder diffraction provides 10 to 20 diffraction lines.
- Background fitting and angular resolution have a strong influence on the validity of powder data analysis while single-crystal data are much less affected by these parameters.

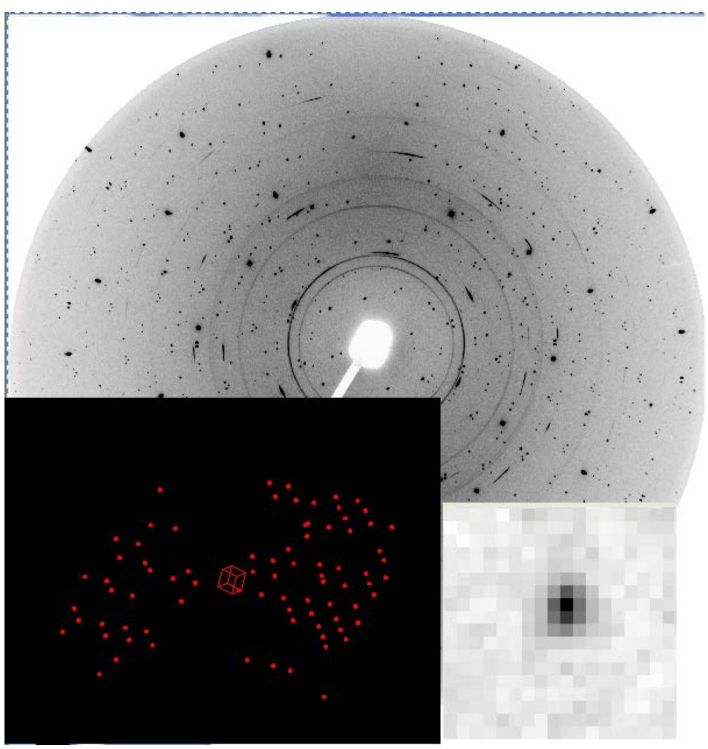
We started a program at the ALS to make beamline 12.2.2 compatible with single-crystal diffraction techniques for diamond cells. We successfully tested the capabilities of the station with regard to single-crystal diffraction and conducted compression studies on suitable samples to mid-mantle pressures (Fig. 1). Subsequently, the ALS adapted the 12.2.2 beamline control interface for single-crystal studies. Moreover, ALS modified the beamline set-up to allow for most efficient reciprocal space sampling. In sum, ALS 12.2.2 should be considered a highly competitive beamline for high-pressure single crystal diffraction studies.

Future plans:

1) **EOS studies on mantle minerals.** Initially at 300 K, later on at temperatures to above 1000 C, we will examine the compression behavior of lower mantle and transition zone minerals as function of composition. This project is directly relevant to the COMPRES community. ALS 12.2.2 has an ongoing project on development of externally heated diamond cells. It will be important to combine reliable external heating over large P-T ranges with the a large aperture necessary for efficient reciprocal space sampling in diffraction experiments.

2) **Extension of SXD to strain analysis in polycrystalline samples.** Many relevant minerals will never be accessible as single crystals; this is true, for example, for post-perovskite which cannot be quenched and reloaded. Moreover, anisotropic stresses between contacting crystals can become an important tool in examining anelastic properties of mantle minerals: Recent efforts [ALS GUP #4456 by Tschauner and Burnley] aim to use time-resolved micro-Laue diffraction at beamline 12.3.2 of the ALS for: a) Establishing stress-strain relations in single crystals of mantle minerals as function of time; b) Examining in-situ dislocation glide and climb, and c) Mapping the variation of stress and strain in polycrystalline aggregates during deformation. The experiments are to be conducted at Earth-like mantle pressures in diamond cells on single-crystal samples, as schematically illustrated in Fig. 2.

3) **Structure analysis from very small grains.** In many cases, it is important to examine the structure of coexisting phases in situ. This holds for non-quencheable phases as well as for in-situ studies of minor element distribution. However, rotation of micron-size crystallites for monochromatic SXD is not feasible while powder diffraction lacks accuracy in quantifying lattice strain. At ALS 12.2.2 X-ray energy can be changed over a range of more than 20 keV without drift in beam-position. This allows for efficient sampling of reciprocal space even for crystallites too small for position conserving sample rotation. This opens a unique opportunity for structure analysis from micron-scale grains in aggregates and for examining minor element-distribution between phases in situ. At ALS 12.3.2 N. Tamura and M. Kunz are developing this energy-scan method of structure analysis from very small grains and we are collaborating with them in examining micrometer scale crystals. ALS 12.2.2 can profit from this effort and adapt this technique for examining multi-phase assemblies in diamond cells.



Presently, P. Dera (U. Chicago), P. Burnley (UNLV), S.M. Clark (LBL), B. Lavina (UNLV), J. Smyth (U. Colorado) and O. Tschauner (UNLV) are using these new single-crystal capabilities at the ALS. P. Dera played an essential role in setting up the SXD program at 12.2.2, ALS.

Figure 1: Wide oscillation pattern and reciprocal space projection of a xenotime single-crystal specimen at 15 GPa. Neon- and diamond diffraction is visible but does not affect indexing. We indexed ~200 reflections and obtained an integral R-factor of 5%. This indicates that the combined effect of circle of confusion, detector calibration, polarization correction and sample-specific parameters such as absorption correction and changes in the diffraction volume can be kept sufficiently low to permit reliable structure analysis. More-over, the number of observed re-flections provides excellent statis-tics for cell parameter determi-nation.

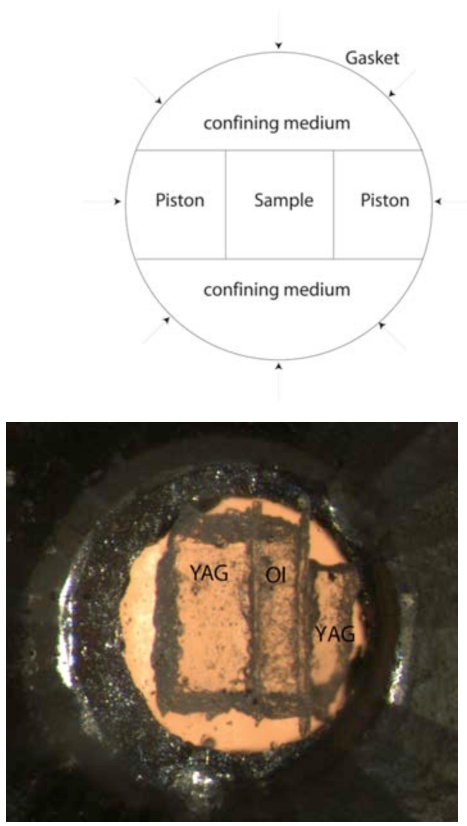


Figure 2: Schematics and example of single crystal deformation DAC sample assembly. Sample is approximately 100 x 100 x 50 microns. Piston crystals will be laser cut with rounded outer edges. As pressure is increased, the creep of the gasket forces the piston crystals closer together and deforms the sample crystal. A liquid confining media eliminates tractions between the sample and the diamond faces and allows for a well defined stress state in the sample.
Initial experiment with Olivine (Ol) and two YAG (YAG) single crystals cut using a micro drill. In the future we will use laser cut samples.

B.3: On-going Community Capabilities Resulting From Previously-Funded COMPRES Initiatives

Many projects initiated and sponsored by COMPRES under the Infrastructure Development program are in operation and, although no longer supported financially by COMPRES, are now available to the community. A list of such projects is given below.

B.3.1 Brillouin Scattering + Synchrotron X-ray diffraction system (13-BM-D, GSECARS, APS)

PIs: Jay Bass, Stas Sinogeikin (Illinois), Guoyin Shen (HPCAT), Vitali Prakpenka, Chicago

This system provides access to single-crystal elasticity measurements for the entire community in a central location. It was the first facility to combine Brillouin spectroscopy and synchrotron X-ray diffraction, providing a unique capabilities for completed characterization of material response. It has now been adopted at other synchrotrons. This facility now attracts between 6-8 users per run cycle at beamline 13 BM-D of GSECARS. It serves as a centralized Brillouin scattering facility that opens up this technique to the entire community, including non-expert users. The facility can be used for conventional Brillouin experiments when there is the synchrotron beam is down. COMPRES purchased equipment and funds for construction and commissioning were leveraged from an Elasticity Grand Challenge grant to Bass (UIUC).

B.3.2 Offline CO_2 laser heating system (GSECARS, APS)

PIs: Thomas Duffy, Princeton, Dion Heinz, Chicago, Guoyin Shen, Carnegie, Vitali Prakapenka, Chicago

An alternative to traditional near-IR laser heating systems, CO_2 laser heating extends the range and types of samples that can be subjected to high P-T conditions. For example, the CO_2 laser (10.6 um) will be absorbed even by Fe-free silicates, and thereby avoids contamination or degradation of signals due to laser absorbing media. CO_2 laser heating is also highly useful in combination with spectroscopic experiments because the sample can be transparent to visible light (e.g. Raman, IR, Brillouin). The CO_2 heating system at GSECARS is unique in its incorporation of an infrared camera for focusing and sample viewing (AGU abstract).

B.3.3 Portable Paris-Edinburgh cell for melt and liquid property determination at high pressures (HPCAT, GSECARS, APS)

PI: Yanbin Wang, Chicago + others?

Structural and related physical properties of non-crystalline materials, i.e., glasses, liquids, and melts at high pressure are of fundamental importance in geophysics. During COMPRES II, an infrastructure development project (in collaboration with GSECARS and HPCAT) has established a new user facility at the Advanced Photon Source for liquid and melt property studies. A portable Paris-Edinburgh (PE) press system has been set-up and extensively tested. This PE press set-up is the only facility in the world aimed at “complete” liquid/melt property characterization by combining X-ray diffraction (for structural characterization), absorption (density), radiographic observation (viscosity), and ultrasonic interferometry (elasticity) measurements. Several silicate melts have been studied to test the capability of the facility. Yamada et al. (2010) studied albite and anorthite to 6 GPa and 2000 K. Sakamaki et al. (2010) studied the melt system along the jadeite (Jd) – diopside (Di) join. Kono et al (2010) tested ultrasonic setup with the PE press. Mei et al (2010) used the PE system for measuring the structure of MgSiO3 glass up to 9 GPa. The facility will be open to general users starting in 2011.

B.3.4 DDIA-30: A new high-pressure apparatus for the COMPRES community (GSECARS, APS)

PI: Yanbin Wang (Chicago)

The DDIA-30 is a new high-pressure device jointly supported by GSECARS and COMPRES that has been installed and tested at sector 13 of the Advanced Photon Source [1]. This is a dual purpose device. As a deformation device, its operational principle is similar to the deformation DIA (D-DIA) [2] but is much larger in size (with anvil truncation edge lengths (TEL) in excess of 30 mm) and hydraulic load capacity of 1000 tons. The upper and lower guide blocks have built-in differential hydraulic rams, so that the upper and lower anvils can be driven independently, generating a controlled differential stress field. When operated in single-stage mode, the device allows large samples (up to ~10 mm) to be deformed under high pressure and temperature, in a way identical to the smaller D-DIA. The large TEL and load capacity makes DDIA-30 more attractive in double-stage configurations. We have tested six DIA anvil extensions with small TELs using monochromatic diffraction and imaging in DDIA-30. This 6-6 configuration [3] allows deformation experiments to be conducted to much higher pressures than in the smaller D-DIA. Recent laboratory studies using a similar device in Japan have shown that the large guide blocks have unique advantages in maintaining anvil alignment, greatly expanding deformation capability to 25 GPa and 2000 K [4]. As an ultra-high pressure device, DDIA-30 is used to compress eight second-stage cubic anvils without driving the differential rams. This 6-8 (6 first-stage and 8 second-stage anvils) configuration has been demonstrated to reach 90+ GPa with sintered diamond as second-stage anvils [5, 6]. We have tested this configuration to 35 GPa and 1500 C and successfully conducted melting experiments on selected metals and alloys with energy-dispersive diffraction and imaging. The new DDIA-30 adds a powerful tool for the large-volume high pressure community.

References

[1] Wang, Y., Z. Jing, N. Hilairet, T. Yu, N. Nishiyama, Y. Tange, T. Sakamaki, M. Rivers, S. Sutton (2010) DDIA-30: a versatile megabar mutlianvil device for in-situ high pressure studies with white and monochromatic synchrotron radiation, MR13A-1908, AGU Fall Meeting, 2010.

[2] Wang, Y., W. B. Durham, I.C. Getting, D.J. Weidner (2003) The deformation-DIA: A new apparatus for high temperature triaxial deformation to pressures up to 15 GPa. Review of Scientific Instruments 74, 3002-3011.

[3] Nishiyama, N., Y. Wang, T. Sanehira, T. Irifune, M. Rivers (2008) Development of the Multi-anvil Assembly 6-6 for DIA and D-DIA type high-pressure apparatuses. High Pressure Research: An International Journal 28(3), 307 - 314.

[4] Kawazoe, T., N. Nishiyama, Y. Nishihara, T. Irifune (2010) Pressure generation to 25 GPa using a cubic anvil apparatus with a multi-anvil 6-6 assembly. High Pressure Research 30(1), 167-174.

[5] Tange, Y., T. Irifune, K. Funakoshi (2008) Pressure generation to 80Â GPa using multianvil apparatus with sintered diamond anvils. High Pressure Research: An International Journal 28(3), 245-254.

[6] Ito E., Yamazaki D., Yoshino T., Fukui H., Zhai S., Shatzkiy A., KatsuraT., Tange Y., Funakoshi K. (2010) Pressure generation and investigation of the post-perovskite transformation in MgGeO3 by squeezing the Kawai-cell equipped with sintered diamond anvils, Earth and Planetary Science Letters, 293, 84-89.

B.3.5 Heating Externally to Extreme Temperatures in the Diamond Anvil Cell (HEETDAC)

Q. Williams (Santa Cruz), H. R. Wenk (Berkeley)

As part of an in-progress infrastructure development project (HEETDAC, funded in FY 2010-2011) and earlier development work carried out at the COMPRES West Coast Synchrotron facility (ALS), a novel diamond anvil cell design and heating technique is being developed that has already resulted in the shattering of past records for the pressure and temperature range of an externally heated diamond anvil cell in radial diffraction geometry. This cell has now accessed the pressure/temperature range up to 40 GPa and 2000 K. This breakthrough is the result of a new heating design utilizing a graphite heater directly juxtaposed with the diamonds and a constant rapid flow of argon. Using this design the diamond anvils survive the high-temperature conditions.

The thermal characteristics of this heater are being refinedto achieve significantly higher temperatures. The high-pressure limits of this assembly have yet to be explored. The prospect here is that the externally-heated diamond cell may achieve a temperature-pressure range approaching those of multi-anvil presses, but with optical access to the sample. This on-going Infrastructure Development Project is optimizing the design and probing the performance limits of this external-heating apparatus and ultimately will make its design (and hence capabilities) accessible to the broader COMPRES community. Simultaneous laser-heating with external heating is also under development to produce lower thermal gradient laser-heated spots. This new capability markedly enhances the range of science that can be conducted using this apparatus. These include utilizing the externally heated diamond cell to study the kinetics of mantle phase transitions, exploring the deformation mechanisms of mantle materials, and conducting petrologic experiments at mid-mantle conditions. Additional information on the resistance-heating project is given above in the ALS section of Part B of this proposal.

B.3.6 Mineral Physics Educational Modules for Advanced Undergraduates and Graduate Students

Pamela Burnley (University of Nevada at Las Vegas)

An in-progress COMPRES project begun in January 2011 is working to assemble a group of web-based educational modules for a course entitled "Introduction to Mineral Physics". The modules are being designed to function as part of a full semester course, although each module is also able to stand alone. The modules are targeted at entry level graduate students and advanced undergraduate students. Learning outcomes for the course have been developed in consultation with educators throughout the COMPRES community. The materials are being designed to be compatible with common distance learning platforms. Potential users include COMPRES members teaching "bricks and mortar" classes at their own institutions, COMPRES members teaching in a distance education setting, mineralogy teachers interested in including supplementary material in their mineralogy class, undergraduates doing independent study projects and graduate students and colleagues in other sub disciplines who wish to brush up on a mineral physics topic. The modules will reside on the SERC "On the Cutting Edge" web site (see below) in the teaching Mineralogy collection and there will be direct links to the materials from the COMPRES web site. The existence of the modules will facilitate the creation of graduate distance education courses in mineral physics that could serve mineral physics graduate students nationwide. The creation of such courses would address current problems faced by faculty in state universities where rising minimum enrollments are making it difficult to teach a suitable graduate course to incoming students. Pamela Burnley of UNLV is leading this project and coordinating the effort with other faculty in other COMPRES institutions.

The above project will contribute to a growing partnership between COMPRES and the *On the Cutting Edge* project supervised by Dovid Mogk of Montana State University as part of the Science Education Research Center (SERC) of Carleton University (see: http://serc.carleton.edu/NAGTWorkshops/mineralogy/mineral_physics.html). Dave Mogk gave a presentation at the 2009 Annual Meeting of COMPRES and interacted with many members of our community. A number of members of our community including Alex Navrotsky (Davis), Mike Brown (Washington), Abby Kavner (UCLA), Pamela Burnley (UNLV), and Artem Oganov (SBU) have made mineral physics-related contributions of educational modules and tools to this website. **(Couldn't find some of these contributions on the webpage?(I couldn't either - I'll ask Dave)**. As another example of our collaboration with the *On the Cutting Edge* professional development program, Wendy Panero (Ohio

State), will be a co-convener for an August 2011 workshop on *Teaching Mineralogy, Petrology, and Geochemistry in the 21st Century* to be held at the University of Minnesota.

Supplementary Information

Membership of COMPRES
Funding History of COMPRES
Relationship between COMPRES and GSECARS
Workshop History
Committee History
Letters of Support
Publications of COMPRES 2006-2011

COMPRES Member Institutions 2011

U. S. Institution	Elector	Alternate
Argonne National Laboratory	Ercan Alp	
Arizona State University	Thomas Sharp	James Tyburczy
Auburn University	Jianjun Dong	
Azusa Pacific University	Donald Isaak	
Brookhaven National Laboratory	Lars Ehm	Markus Hücker
California Institute of Technology	Jennifer Jackson	Paul Asimow
Carnegie Institution of Washington	Ronald Cohen	Yingwei Fei
Case Western Reserve University	James Van Orman	Nancy Chabot
Colorado College	Phillip Cervantes	
Columbia University	David Walker	Taro Takahashi
Cornell University	William Bassett	Zhongwu Wang
Delaware State University	Gabriel Gwanmesia	Al Sameen Khan
Florida International University	Jiuhua G. Chen	Surendra Saxena
Harvard University	Sarah Stewart-Mukhopadhyay	Richard O'Connell
Indiana University at South Bend	Henry Scott	Jerry Hinnefeld
Johnson Space Center , NASA	Kevin Righter	John Jones
Lawrence Berkeley National Laboratory	Simon Clark	Corwin Booth
Lawrence Livermore National Laboratory	Daniel Farber	
Los Alamos Natoional Laboratory	Dana Dattelbaum	Jianzhong Zhang
Louisiana State University	Bijaya Karki	
Massachusetts Institute of Technology	San-Heon (Dan) Shim	Robert van der Hilst
New Mexico State University	Boris Kiefer	
Northern Illinois University	Mark Frank	Jonathan Berg
Northwestern University	Steven Jacobsen	Craig Bina
Ohio State University	Wendy Panero	Michael Barton
Princeton University	Thomas Duffy	Frederik Simons
Rensselaer Polytechnic Institute	Liping Huang	Bruce Watson
Smithsonian Institution	Elizabeth Cottrell	Jeffrey Post
Stanford University	Wendy Mao	Jonathan Stebbins
Stony Brook University	Michael Vaughan	John Parise
Texas A&M University	Caleb Holyoke	
Texas Tech University	Yanzhang Ma	Valery Levitas
University of Arizona	Robert Downs	Michael Drake
University of California at Berkeley	Hans-Rudolph Wenk	Raymond Jeanloz
University of California at Davis	Charles Lesher	Alexandra Navrotsky
University of California at Los Angeles	Abby Kavner	Donald Isaak
University of California at Riverside	Harry Green	Stephen Park
University of California at San Diego	Guy Masters	
University of California at Santa Cruz	Quentin Williams	Elise Knittle

University of Chicago	Dion Heinz	Mark Rivers
University of Colorado at Boulder	Joseph Smyth	Hartmut Spetzler
University of Hawaii at Manoa	Murli Manghnani	Li Chung Ming
University of Illinois at Urbana-Champaign	Craig Lundstrom	
University of Louisville	George Lager	
University of Maryland at College Park	Wenlu Zhu	John Tossell
University of Michigan	Rebecca Lange	Youxue Zhang
University of Minnesota	Renata Wentzcovitch	Tony Withers
University of Missouri - Kansas City	Michael Kruger	Ray Coveney
University of Nevada at Las Vegas	Oliver Tschauner	Pamela Burnley
University of New Mexico	Carl Agee	Penny King
University of Texas at Austin	Jung-fu Lin	Stephen P. Grand
University of Washington	Michael Brown	
University of Wyoming	David Anderson	
Virginia Polytechnic Institute and State University	Nancy Ross	Ross Angel
Washington University in St Louis	Phil Skemer	
Yale University	Shun-ichiro Karato	Kanani Lee

COMPRES Foreign Affiliates

Australian National University Canberra (Australia)	Hugh O\'Neill
Bayreuth Universitat (Germany)	David Rubie
Chinese Academy of Science (China)	Changqing Jin
China University of Geosciences of Wuhan (China)	Zhenmin Jin
Ecole Normale Supérieure de Lyon (France)	Jan Matas
Ehime University (Japan)	Tetsuo Irifune
Eidgenossische Techniche Hochschule Zurich (Switzerland)	Carmen Sanchez-Valle
GeoForschungsZentrum Potsdam (Germany)	Hans-Joachim Mueller
Harbin Institute of Technology (China)	Haozhe Liu
Institut de Physique du Globe Paris (France)	Guillaume Fiquet
Institute for Earth's Crust, Irkutsk (Russia)	Peter Dorogokupets
Institute of Experimental Mineralogy, Chernogolovka (Russia)	Yuriy Litvin
Jilin University (China)	Xiaoyang Liu
Macquarie University Sydney (Australia)	Tracy Rushmer
Max-Planck Institute for Solid State Research, Stuttgart (Germany)	Thomas Locherer
National Cheng Kung University (Taiwan)	Jennifer Kung
Novosibirsk State University (Russia)	Elena Boldyreva
Okayama University (Japan)	Eiji Ito
Peking University (China)	Qiong Liu
Royal Institution of Great Britian, The (United Kingdom)	Paul McMillian
Ruhr-Universitat Bochum (Germany)	Sumit Chakraborty
Seoul National University (Korea)	Haemyeong Jung
Tel Aviv University (Israel)	Moshe Pasternak
Tohoku University, Sendai (Japan)	Eiji Ohtani
Universitat Frankfurt am Main (Germany)	Bjorn Winkler
Universite Blaise Pascal (France)	Denis Andrault
Universite de Poitiers (France)	Jacques Rabier
Universite des Science et Technologies de Lille (France)	Paul Raterron
Universite Paul Sabatier (France)	Jannick Ingrin
University College London (United Kingdom)	David Dobson
University of Calgary (Canada)	Sytle Antao
University of Cambridge (United Kingdom)	Michael Carpenter
University of Edinburgh	Geoffrey Bromley
University of Manchester (United Kingdom)	Alison Pawley
University of Tokyo (Japan)	Takehiko Yagi
University of Wales at Aberystwyth (United Kingdom)	Martin Wilding
University of Western Ontario (Canada)	Rick Secco
Vrije Universiteit (The Netherlands)	Wim van Westrenen
Yonsei University (Korea)	Yongjae Lee

Funding History of COMPRES & Current-Year Budget

(All in units of $1K)

May 1 2002 to April 30 2007: First Cooperative Agreement

Year 1	Year 2	Year 3	Year 4	Year 5	Cumulative
1967	2200	2227	2100	2100	10594

June 1 2007 to May 31, 2012: Second Cooperative Agreement

Year 1	Year 2	Year 3	Year 4	Year 5	Cumulative
2100	2100	2100	2400	2400	11100

An ARRA supplement in Year 3 was granted for $850K in Equipment

Current Year #5 of COMPRES, 2nd C.A.

	Facilities		
NSLS DAC XRD	Ops	337.5	
	Equip	40	
NSLS MAP	Ops	404	
	Equip	21	
NSLS U2A	Ops	220	
	Equip	14	
ALS 12.2.2	Ops	249	
	Equip		
Infrastructure Development			
Education Modules		61.8	UNLV
GSECARS, Gas Loading		38.7	U Chicago
IXS at the APS (Alp)		76.1	Sector 3, Nuclear Resonant Scattering
Multi-anvil cell assemblies		78.8	ASU
Elasticity Database		16.2	UIUC
FIB/SEM nanofabrication		25	Workshops/Training, Geophysical Lab
Subawards IDC		0.4	
Annual Meeting		100	
Travel, Committees		33	
Lecture Series		20	
Beamline Housing		35	
Workshops		90	
PUP Sector 3 (Detector)		60	
Central Office	Salaries + Fringe +IDC	424.4	
	Supplies	22	
	Travel	35	
	Web Service	3	
Total		2400	

Relationship of GSECARS and COMPRES

GeoSoilEnviroCARS (GSECARS), at the University of Chicago. is a national user facility for research in the Earth sciences using the third generation Advanced Photon Source (APS), Argonne National Laboratory. GSECARS and COMPRES are complementary organizations that collaborate closely through coordination of community development activities, and the design, construction and operation of advanced instrumentation. 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, and other young researchers in the Earth science community. All beam time at GSECARS and at 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. 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. There have been a number of COMPRES Infrastructure Development Projects that are located, at least in part, at GSECARS. Examples include: (1) a Brillouin spectroscopy system (2) a gas-loading facility for diamond-anvil cells, and (3) D-DIA 30 deformation module for the large-volume press. Optics and software developed at GSECARS are being used at the COMPRES-operated X-ray beamlines at the NSLS. Przemek Dera, Senior Research Associate at GSECARS, is an elected member of the COMPRES Executive Committee. GSECARS participates in the COMPRES annual meeting with presentations on overall GSECARS status and plans, as well as COMPRES Infrastructure projects that are located at GSECARS. The COMPRES Facilities Committee visits GSECARS and provides advice to its directors on current operations and future directions. The Executive Director of COMPRES is a member of the Board of Governors of the Consortium for Advanced Radiation Sources (CARS), of which GSECARS is a part.

We anticipate this cooperation to continue during the upcoming GSECARS and COMPRES renewal periods. GSECARS staff are members of the teams that submitted successful proposals for two future high-pressure x-ray beamlines at NSLS-II at Brookhaven. The timing of construction of these beamlines and the role that COMPRES will play in these facilities is still under consideration at this time. It is hoped that NSF and DOE will contribute resources to permit this new source to be optimally used by the earth sciences community.

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. That document is the basis for this statement.

COMPRES Workshop History

	2011	
Oct 4	ALS user meeting workshop: Single-Crystal Diffraction under Extreme Conditions	ALS
Sep 1 -2	FIB/SEM Training	Carnegie Inst of Washington
Jun 19 - 21	Focused Ion Beam (FIB)/SEM Techniques	Carnegie Inst of Washington
Jun 16	Tutorial: Introduction to Neutron Scattering (Chris Tulk)	COMPRES Annual Meeting at Kingsmill Resort, VA
Jan 24 - 28	Dynamic Phenomena under Extremes	UT Austin
	2010	
Dec 11	Lujan Workshop: Applications of Neutron Scattering to Materials and Earth Sciences	UC Berkeley
Aug 30-31	Computational Infrastructure For Mineral Physics: A Community Consultation Workshop.	Univ of Minnesota, Minneapolis, MN
Jun 15 - 16	Time-Resolved X-ray Diffraction and Spectroscopy at Extreme Conditions (TEC): A High-Pressure Beamline Workshop for the National Synchrotron Light Source II (NSLS II)	NSLS, BNL
Apr 29 - 30	4-Dimensional Studies in Extreme Environments (4DE): A High- Pressure Beamline Workshop for the National Synchrotron Light Source II (NSLS II)	NSLS, BNL
	2009	
Dec 12 - 13	Laser Heating the DAC: Where we are and where we are going	ALS of the Lawrence Berkeley National Lab
Sep 23 - 25	On-line Brillouin Spectroscopy at GSECARS: Basic Principles and Application for High Pressure Research	APS, ANL, Argonne
Jul 25 - 30	American Crystallographic Association Meeting	Toronto, Ontario, CANADA
May 17 - 20	CIDER '09 Community Workshop	Marconi Center, CA
Mar 2 - 4	Long-Range Planning Workshop for High Pressure Earth Sciences	Fiesta Resort Conference Center, Tempe, AZ
	2008	
Oct 4	NSLS Workshop: Advances in High-Pressure Science Using Synchrotron X-rays	NSLS
Jun 22 - Aug 8	The Third Summer CIDER Program	Kavli Institute of Theoretical Physics, Santa Barbara, CA
May 31 - Jun 1	Workshop to Introduce High-Resolution Inelastic X-ray Scattering on Earth Materials using Synchrotron Radiation.	Advance Photon Source, ANL
Jan 22 - 23	Earth and Environmental Sciences	NSLS

Jan 17-18	MS&E: Materials at High Pressure	NSLS
Jan - Mar	Planning Workshops for NSLS and NSLS-II	NSLS
Jan - Mar	Scientific Strategic Planning Workshops	NSLS
	2007	
Dec 6 - 7	International Workshop on Synchrotron High-Pressure Mineral Physics and Materials Science	APS, ANL
Sep 3 - 7	Workshop on Advances in High Pressure Crystallography at Large Scale Facilities	Wadham College, Univ of Oxford
Aug 5 - 10	The second VLab workshop - Minnesota Supercomputing Institute	Minneapolis, MN
Jul 9 - 29	"Many-body theory of inhomogeneous superfluids" - Pisa, Italy, at Centro di Ricerca Matematica "Ennio De Giorgi"	Scuola Normale Superiore, ScopeCentre
Mar 15 - 16	Workshop on Calorimetry-on-a-Chip	Univ of California at Berkeley
Jan 26 - 28	Workshop on Current Status and Prospects for Establishing Precise and Accurate Pressure Scales at High Temperatures	Geophysical Lab of the Carnegie Inst of Washington
	2006	
Dec 7-8	Mineralogical Society of America Short Course Neutron Scattering in Earth Sciences	Berkeley
Sep 3 - 7	Rheology Grand Challenge Workshop	Stony Brook Univ
Aug 5 - 10	Half-day session on Non-Ambient Crystallography -- ACA 2006 Annual Meeting on Monday afternoon, July 24, 2006	Honolulu, Hawaii
Jun 17 - 21	Seismic Anisotropy and Geodynamics of the Lithoshpere - Asthenosphere System	Chateau of Trest, Czech Republic (southern Moravia)
May 23 - 26	"M03: Spectroscopy in Mineralogy: Theory and Experiment" - 2006 Joint Assembly Meeting	Baltimore MD
Apr 29 - May 1	Workshop on Synergy of 21st Century High-Pressure Science and Technology - Advanced Photon Source	ANL, Argonne
Apr 29 - May 1	Synergy of 21st Century High-Pressure Science and Technology	APS
Apr 28	Working Group on Gas-Loading DAC system at APS	APS
Mar 2 - 7	Frontiers in High Pressure Research A German-Israeli Minerva School	Kibbutz Ein-Guedi, Dead-Sea, ISRAEL
Feb 25 - 26	Workshop on Future Directions for X-ray High-Pressure at the NSLS (X-Ray High Pressure Research Workshop: Current operation and vision into NSLS II)	NSLS
No info	MSA Short Course on Water in Nominally Anhydrous Minerals	Berkeley
No info	Marie Curie Summer School 2006- Composite & Polyphase Materials	Italy

History of COMPRES Committee Membership

Executive Committee

Last Name	First Name	Member	Chair	Vice Chair	President
Agee	Carl	2007-2009		2008-2009	
Bass	Jay	2002-2008			2002; 2010-present
Brown	Michael	2005-2007		2007	
Dera	Prezemek	2010-2013			
Duffy	Thomas	2002; 2010-2013	2010-2013		
Green	Harry		2004-2006		
Hemley	Russell	2002-2003			
Karato	Shun			2002-2004	
Liebermann	Robert				2003-2009
Ross	Nancy	2010-2013			
Tyburczy	James	2008-2014			
van Orman	James	2009-2012		2010-2012	
Weidner	Donald	2002-2009	2002-2003		
Williams	Quentin	2004-2009	2007-2009	2004-2006	

Community Facilities Committee

Last Name	First Name	Member	Chair
Brown	Michael	2002	
Campbell	Andrew	2008-2014	2011-2013
Duffy	Thomas	2003-2005; 2007-2009	2007-2008
Durham	William	2005-2007	
Fei	Yingwei	2002-2004	
Green	Harry	2002-2003	
Jackson	Jennifer	2010-2013	
Kavner	Abby	2004-2006	
Lesher	Charles	2006-2008	
Mao	Wendy	2009-2012	
Panero	Wendy	2006-2012	2009-2011
Prewitt	Charles	2003-2005	
Rivers	Mark	2002-2007	2003-2006
Wang	Yanbin	2008-2014	
Williams	Quentin		2002

Infrastructure Development Committee

Last Name	First Name	Member	Chair
Burnley	Pamela	2002-2007; 2010-2013	2010-2012
Hemley	Russell	2005-2007	
Jacobson	Steven	2008-2014	
Jeanloz	Raymond	2002	
Kavner	Abby	2009-2014	
Knittle	Elise	2011-2014	
Li	Jie	2008-2010	
Righter	Kevin	2003-2005	
Ross	Nancy	2003-2008	2004-2007
Sharp	Thomas	2006-2010	2008-2009
Shim	Dan	2004-2010	
Tschauner	Oliver	2010-2013	
Tyburczy	James	2002-2003	2002-2003
Walker	David	2002	
Wang	Yanbin	2002-2004	

Advisory Council

Last Name	First Name	Member	Memo
Jephcoat	Andrew	2009-2012	
Buffett	Bruce	2002-2006	
Chen	Wang-ping	2006-2008	
Garnero	Edward	2008-2010	
Heaney	Peter	2009-2012	
Kao	Chi-chang	2002-2010	
Kellogg	Louise	2007-2013	
Lay	Thomas	2011-2014	
Masters	Guy	2002-2007	
McDonough	William	2007-2010	
Nicol*	Malcolm	2006-2008	
O'Connell	Richard	2002-2006	
Silver*	Paul	2002-2005	
Romanowicz	Barbara	2011-2014	
Van Keken	Peter	2010-2013	

* deceased

Letters of Support: NSLS-II and APS

Photon Sciences Directorate

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for the U.S. Department of Energy

August 1, 2011

Prof. Jay Bass
President of COMPRES
Ralph E. Grimm Professor of Geology
Department of Geology
University of Illinois, Urbana - Champaign
208 Natural History Building
1301 W. Green St.
Urbana, IL 61801

Dear Prof. Bass,

I am writing to enthusiastically express my strongest possible support for your renewal proposal to the NSF Earth Sciences Division for continued support of COMPRES at the National Synchrotron Light Source and in the future at the National Synchrotron Light Source II. Our partnership in operating beamlines X17 (with end stations X17B2, X17B3, and X17C) and U2A has made them among the most productive beamlines at the facility. These beamlines are routinely oversubscribed by up to a factor of 7 and consistently produce more than 70 publications a year, with many in high-impact journals. Importantly, they are the workhorse beamlines for the high pressure earth sciences user community, supporting dozens of user groups per year.

This success can be attributed to the state-of-the art beamlines, unique endstation capabilities, and the outstanding scientific and technical support provided by COMPRES to the users. To cite just one example, COMPRES has pioneered the advanced tools necessary to move the realm of rheology measurements from the 100's of MPa to 10's of GPa. Your contributions have enabled us to benefit from the expertise of your scientific staff and to achieve higher operational efficiency by standardizing instrumentation and software across the beamlines at NSLS used by the high pressure earth sciences user community. The technologies developed by COMPRES, and the sample preparation laboratory you maintain, have also benefited the entire high pressure earth sciences user community at NSLS.

As you are aware, NSLS will continue to operate until NSLS-II begins operations, which is currently planned for mid-FY14. We envision thriving earth sciences and high pressure sciences communities at NSLS-II that fully capitalizes on its significantly enhanced capabilities. We look forward to continuing our very productive partnership with COMPRES to help maintain the current strong research environment and to foster new science programs at NSLS-II.

We are working to establish best in class beamlines and user support for them from the beginning of operations of NSLS-II. Several beamlines under development for NSLS-II will enable the user community to address some of the most pressing problems in earth sciences and high pressure sciences. COMPRES is playing a critical role in the development of a particularly important one of these, i.e., the X-ray Powder Diffraction beamline (XPD).

XPD is designed to be a high-energy, high-resolution powder diffraction beamline for in situ and/or in operando investigation of materials in extreme environments. We look forward to a partnership with COMPRES in operating XPD. Your transfer of the existing equipment and expertise in multi-anvil and diamond anvil high pressure systems at NSLS to XPD will complement the extreme environments at XPD with world class high pressure capabilities and COMPRES staff will provide the high quality of user support that will enable the most challenging problems to be done. Since the XPD beamline will be operational from the very outset of NSLS-II operations, this arrangement assures that COMPRES programs will have a smooth transition from NSLS and be supported from the very start of NSLS-II.

Several other beamlines that are currently under construction, such as HXN (Hard X-ray Nanoprobe) and IXS (Inelastic X-ray Scattering), will also provide unprecedented experimental capabilities for the earth sciences and high pressure community. These beamlines exploit the outstanding capabilities that the new light source will offer for high energy resolution and high spatial resolution and will also commence operations at the very start of NSLS-II.

We look forward to further developing our partnership with COMPRES to make a wide range of high pressure synchrotron research tools available to the user communities at these beamlines and with the high quality of user support that will enable them to carry out the most challenging problems. We thus welcome the creation of a COMPRES science coordinator at NSLS-II, to provide and disseminate valuable expertise in high pressure sciences and instrumentation to these and other beamlines at NSLS-II and to enhance the overall scientific portfolio of NSLS-II in research at extreme conditions.

In addition to XPD and the other beamlines currently under construction mentioned above, we look forward to working closely with COMPRES to seek funding to build additional beamlines needed by the earth sciences and high pressure communities. We have already received several proposals for such beamlines from the user community and after strenuous peer review the most compelling of them have been approved for construction by NSLS-II management and our Science Advisory Committee, pending identification of the necessary funding.

COMPRES played a leadership role in several of these successful proposals. In particular, FIS (Frontier Synchrotron Infrared Spectroscopy Beamline under Extreme Conditions) will specialize in high-pressure IR studies. NSLS-II will provide world leading brightness and flux in the infrared and FIS will enable the strong NSLS program at U2A to continue at NSLS-II. 4DE (4-D Studies in Extreme Environments) and TEC (Time-resolved X-ray Diffraction and Spectroscopy under Extreme Conditions) are insertion-device based beamlines for cutting-edge large-volume and diamond cell high pressure research. These beamlines will dramatically expand the reach of the already strong large-volume and diamond cell programs at X17 and form the foundation of a world class high-pressure earth science research capability at NSLS-II. All of these are key to achieving our vision of thriving earth sciences and high pressure sciences communities at NSLS-II. As such, we consider them to have a very high priority and we are fully committed to working with COMPRES to secure the funding needed for their construction.

We look forward to the continued engagement of, and partnership with, COMPRES, in NSLS today and in NSLS-II in the future, in order to ensure a smooth transition and continued strong support for the earth sciences and high pressure sciences communities that takes maximum advantage of the superlative capabilities that NSLS-II will enable.

You have my very best wishes for a successful proposal and I look forward to continuing our close and productive working relationship for many years.

Sincerely,

Steve Dierker

Steve Dierker
Associate Laboratory Director for Photon Sciences
Director, National Synchrotron Light Source II
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August 22, 2011

Prof. Jay D. Bass
President, Consortium for Materials Properties Research in Earth Sciences
University of Illinois at Urbana-Champaign
Urbana, IL 61801

Dear Prof. Bass,

I am happy to write a letter of support for your proposal to establish COMPTECH at the Advanced Photon Source.

I'd like to thank you and your team for visiting APS last week to discuss in person the COMPTECH concept and COMPRES's interest in augmenting beamline capabilities at APS for high-pressure Earth sciences experimentation. This proposal is an excellent plan to build capabilities, increase efficiency, and expand the APS user community in this area.

In anticipation of a positive outcome of the proposal, we will plan to provide office space at APS for the COMPRES Technology Officer. We look forward to work with COMPRES through Partner User Proposals you plan to submit. As is standard for this type of proposal, the investments in equipment and personnel by COMPRES will be matched by access to beamtime to develop new capabilities at selected APS beamlines, subject to approval by our Proposal Review Panel and Scientific Advisory Committee.

We look forward to building upon the fruitful relationship we have had with COMPRES to date. The significant investments that COMPRES has made in personnel support at Sector 3 over the last 8 years, and in experimental facilities permanently installed at GSECARS, have enabled new capabilities at these beamlines for the entire community and, at Sector 3, has resulted in a much broader high-pressure Earth science user base.

Sincerely yours,

Brian Stephenson

Brian Stephenson
Interim Associate Laboratory Director, Photon Sciences
Argonne National Laboratorys

COMPRES Publications 2006-2011

COMPRES Publications 2006

Facilities

West Coast Synchrotron (Advanced Light Source 12.2.2)

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X-ray DAC Facility (NSLS X17C and X17B3)

Aksoy, R., Y. Ma, E. Selvi, M. C. Chyu, A. Ertas, and A. White (2006), X-ray diffraction study of molybdenum disulfide to 38.8 GPa, *Journal of Physics and Chemistry of Solids*, *67*(9-10), 1914-1917.

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Ross, N. L. (2006), Single-crystal neutron diffraction: Present and future applications, in *Neutron Scattering in Earth Sciences*, vol. 63, edited by H. R. Wenk, pp. 59-80, Mineralogical Society of America, Washington, DC. (Neutrons)
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Higo, Y., T. Inoue, B. Li, T. Irifune, and R. Liebermann (2006), The effect of iron on the elastic properties of ringwoodite at high pressure, *Physics of the Earth and Planetary Interiors*, *159*(3-4), 276-285. **(COMPRES Central)**

COMPRES Publications 2007

Facilities

West Coast Synchrotron (Advanced Light Source 12.2.2)

Caldwell, W. A. et al. (2007), Laser-heated diamond anvil cell at the advanced light source beamline 12.2.2, *Nuclear Instruments and Methods in Physics A, 582*(1), 221-225.
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Diamond Anvil Infrared Facility (NSLS U2A)

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X-ray Large Volume Press Facility (NSLS X17B2)

Botez, C. E., J. D. Hermosillo, J. Zhang, J. Qian, Y. Zhao, J. Majzlan, R. R. Chianelli, and C. Pantea (2007), High-temperature phase transitions in CsH_2PO_4 under ambient and high-pressure conditions: A synchrotron x-ray diffraction study, *J. Chem. Phys.*, *127*(19), 194701.
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Infrastructure Development Projects

GSECARS/COMPRES Gas Loading System

Jacobsen, S. D., C. M. Holl, K. A. Adams, R. A. Fischer, E. S. Martin, C. R. Bina, J. Lin, V. B. Prakapenka, A. Kubo, and P. Dera (2008), Compression of single-crystal magnesium oxide to 118 GPa and a ruby pressure gauge for helium pressure media, *American Mineralogist*, *93*(11-12), 1823-1828.

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Other Infrastructure Development Projects

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COMPRES Publications 2009

Facilities

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Choi, C. L., K. J. Koski, S. Sivasankar, and A. P. Alivisatos (2009), Strain-dependent photoluminescence behavior of CdSe/CdS nanocrystals with spherical, linear, and branched topologies, *Nano Letters*, *9*(10), 3544-3549.
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Marquardt, H., S. Speziale, H. J. Reichmann, D. J. Frost, and F. R. Schilling (2009), Single-crystal elasticity of $(Mg_{0.9}Fe_{0.1})O$ to 81 GPa, *Earth and Planetary Science Letters*, *287*(3-4), 345-352. (Brillouin scattering at Advanced Photon Source)
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<u>COMPRES Publications 2010</u>

<u>Facilities</u>

West Coast Synchrotron (Advanced Light Source 12.2.2)

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X-ray Large Volume Press Facility (NSLS X17B2)

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X-ray DAC Facility (NSLS X17C and X17B3)

Chen, Z., T. Tyson, K. Ahn, Z. Zhong, and J. Hu (2010), Origin of the non-linear pressure effects in perovskite manganites: Buckling of Mn–O–Mn bonds and Jahn–Teller distortion of the MnO_6 octahedra induced by pressure, *Journal of Magnetism and Magnetic Materials*, *322*, 3049-3052.
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