COMPRES

The Consortium For Materials Properties Research in Earth Sciences (IV)
National Facilities and Infrastructure Development for High-Pressure Geosciences Research

Proposal to NSF 2017-2022 PROJECT DESCRIPTION

Consortium for Materials Properties Research in Earth Sciences (COMPRES): National Facilities and Infrastructure Development for High-Pressure Geosciences Research (2017-2022)

Submitted to
National Science Foundation
Division of Earth Sciences
Instrumentation and Facilities Program
September 2, 2016

COMPRES is a community-based consortium charged with the oversight and guidance of user facilities that are vital to our mission of understanding the behavior of materials at the conditions of Earth and planetary interiors. COMPRES facilitates the operation of user facilities and the development of new technologies for high-pressure research, and serves as an alliance and advocate for researchers in high pressure mineral physics and related fields.

This proposal is submitted by COMPRES on behalf of the Executive Committee, which represents the 68 U.S. universities and research institutions that are members of COMPRES. This proposal evolved through an extensive process involving a 2014 planning meeting, the 2015 and 2016 COMPRES annual meetings, a town hall meeting at the 2015 Fall AGU meeting, and public solicitations for new initiatives. This process has allowed the COMPRES community to provide input on the form and content of this proposal. All community input and proposals were reviewed and discussed by the COMPRES standing committees, with decisions on the proposed projects and budgets voted on by the Executive Committee. The materials provided by PIs of COMPRES facilities and infrastructure development projects include input from the numerous faculty members and researchers at U.S. institutions who are engaged in research projects at COMPRES facilities.

The proposal and supporting materials include the Project Description (authorized to deviate from the GBG page limit) and two appendices. The Project Description is written to be a self-contained proposal. More detailed descriptions of the Facilities and the Education Outreach Infrastructure and Development (EOID) projects that represent the main activities of COMPRES can be found in Appendix A, in case the reader wants additional information on a particular facility or project. Appendix B contains 143 one-page summaries of published work made possible by COMPRES facilities or projects during the past five years (2012-2016). The "one-pagers" in this compilation were selected from submissions by the COMPRES community for inclusion in this proposal.

The Project Description gives an overview of the COMPRES community and our shared user facilities, followed by a section on Results from Prior NSF Support, summarizing some of the scientific and technical accomplishments facilitated by COMPRES. The Project Description also includes a description our vision for activities over the next five years to support state-of-the-art research in mineral physics, and a brief outline of our funding request. Budget tables and a summary are also included in the Project Description.

President

Carl Agee	University of New Mexico

Executive Committee

Abby Kavner	University of California, Los Angeles (Chair)
Wendy Panero	Ohio State University (Vice-Chair)
Steve Jacobsen	Northwestern University
Baosheng Li	Stony Brook University
Jie (Jackie) Li	University of Michigan

Facilities Committee

Mark Rivers	University of Chicago (Chair)
Bin Chen	University of Hawaii
Arianna Gleason	LANL/Stanford University
Anne Pommier	Scripps, UC San Diego
Sang-Heon (Dan) Shim	Arizona State University

Education, Outreach, Infrastructure and Development Committee (EOID)

Heather Watson	Union College (Chair)
Jiuhua Chen	Florida International University
Katherine Crispin	Pennsylvania State University
Gabriel Gwanmesia	Delaware State University
Joseph Smyth	University of Colorado

For a complete history of COMPRES committee membership, see:
http://compres.us/about-us/committees

Project Summary

Interpreting geological, geophysical and geochemical observational data in terms of the current state, dynamics, and evolution of the Earth requires knowing the properties of Earth materials—including solids and melts, metals and oxides —at the pressures, temperatures, and chemical complexity of materials that make up planetary interiors. The Consortium for Materials Properties Research in Earth Sciences (COMPRES) is an organization that develops state-of-the-art technologies and facilitates access to community user resources for Earth materials research. This proposal reports on the advances and achievements of COMPRES over the previous 4.5 years (June 2012 – September 2016), and describes the science goals and themes that drive the instrument and facility requirements of our community for the upcoming five years (June 2017 – May 2022). This period offers exciting opportunities as new and upgraded synchrotron facilities will dramatically improve experimental capabilities and allow for novel experiments needed to test hypotheses and address yet-unanswered questions on the nature of deep planetary interiors.

Intellectual merit

COMPRES enables new measurements of material properties that help address a broad scope of fundamental questions in Earth science and new discoveries of materials and properties that transform the way we view the Earth's dynamic behavior. Measurements of elastic properties of Earth materials—especially at the combined extreme pressures and temperatures of Earth's interior—aid in interpreting increasingly high-resolution seismological models in terms of variations in Earth's composition and structure. Frontier measurements of transport properties of materials—including rheology, electrical conductivity, and thermal transport properties—provide keys to understand the large-scale dynamic processes governing the Earth's mechanical flow, chemical mixing, and thermal evolution. On the near horizon, user facilities in frontier infrared spectroscopy will enable measurements of the behavior of volatiles in minerals to elucidate the deep-Earth participation in the carbon and water cycles. Petrology and geochemistry at extreme pressures and temperatures—including measurements of melting behavior, phase equilibria, major element composition, and trace element and isotope partitioning via a spectrum of in situ and post mortem micro-chemical sampling techniques —is providing new information about the behavior of rocks that comprise the deep mantle to interrogate its current state and to test hypothesis about the formation and evolution of the Earth's core-mantle system. Synchrotron-based nuclear scattering measurements are determining new sets of properties for iron and its alloys and melts in order to understand the core's role in the Earth's coupled thermal and chemical evolution.

Broader Impacts

COMPRES provides for (1) operation and support of dedicated beamlines for high-pressure research at national synchrotron facilities; (2) development of new technologies for mineral physics research; (3) training mineral physics and geophysics research communities in data acquisition, analysis, and scientific applications of user facilities; (4) education and outreach programs; with 1-4 in combination leading to new career opportunities for the next generation scientists in both academia and industry in the United States. COMPRES-funded beamline scientists and other technical personnel are the most important ingredient enhancing user experience and scientific productivity at our community facilities. COMPRES user facilities are open to researchers at all levels from educational and research institutions. Our outreach programs are designed to attract and nurture scientific talent within geophysics and mineral

physics, and to maximize the scientific impact of our research. Examples of outreach activities include the COMPRES annual meeting, multiple workshops each year, educational development projects, and the COMPRES Distinguished Lecturer Series. The COMPRES Central Office coordinates all of these activities, maintains a newly designed, dynamic website, and provides cohesion and focus for community priorities and future plans. COMPRES supports and promotes the advancement of students, postdocs, and other young scientists through making facilities available for their research, educational programs, community activities, and employment opportunities. The results of research and technology development facilitated and supported by COMPRES reach far beyond the Earth Sciences, with significant contributions to planetary science, materials science, and condensed matter physics and chemistry.

Project Description Table of Contents

Project Description

1. Introduction

We present in this proposal an overview of COMPRES including 1) its main activities and organizational structure; 2) the accomplishments of COMPRES during the past five years; and 3) the vision, motivation, and work plan for the projects to be carried out over the next five years.

This proposal requests continued NSF support of COMPRES activities for a five-year period from June 2017 to May 2022. We propose to continue our support of innovative high pressure Earth materials research at three national synchrotron facilities: Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory, Advanced Photon Source (APS) at Argonne National Laboratory, and National Synchrotron Light Source-II (NSLS-II) at Brookhaven National Laboratory. We propose that COMPRES also continue its support of the "offline" (meaning not on a synchrotron beamline) Multi-anvil Project at Arizona State University (ASU). In our five-year plan COMPRES will be agile in responding to new opportunities such as participating in the planning and establishment of a Large Volume Multi-anvil Press (LVMAP) user-facility in the U.S., and by increasing support for the Frontier Infrared Spectroscopy (FIS) Beamline being built at NSLS-II which will provide world-leading spectroscopic measurements of minerals under extreme conditions in the diamond anvil cell.

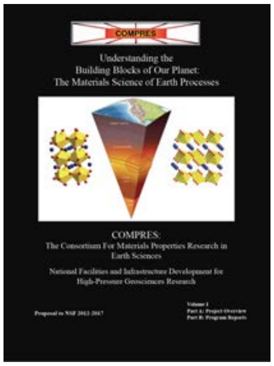
We propose to continue the COMPRES Education Outreach and Infrastructure Development (EOID) program, which seeds projects to develop new instruments, methods, and services for the COMPRES community. We also propose a new significant education and outreach project from Stony Brook University: "A Career Path for African-American Students from HBCUs to National Laboratories". Finally, we request support to continue our successful array of COMPRES-sponsored workshops, meetings, educational initiatives, and outreach programs. The broad range of activities we propose have been defined and vetted by the COMPRES community, and they reflect a collective view of priorities of the high-pressure Earth science community for the future.

The Project Description of this proposal is written as a self-contained proposal, with sufficiently detailed descriptions of each aspect of COMPRES to evaluate the consortium as a whole, our various facilities and infrastructure projects, and our plans for the next five years. Appendix A contains more-detailed descriptions of each COMPRES facility, with additional technical details. In Appendix B, we present a theme-sorted collection of 1-page summaries of COMPRES supported, published research since 2012.

2. COMPRES Summary—Governance, Science, Portfolio

COMPRES, acronym for the **CO**nsortium for **M**aterials **P**roperties **R**esearch in the **E**arth **S**ciences, supports and advocates for research on the materials properties of Earth and planetary interiors with a particular emphasis on high-pressure science, technology, and related fields. COMPRES is charged with the oversight and guidance of several high-pressure laboratory-based open user-facilities, many in conjunction with national synchrotron light sources. These have become not just important tools in Earth science research, but essential facilities for carrying out the research activities for a large portion of the mineral physics community. COMPRES supports the operation of beamlines, develops and maintains user technologies for high-pressure research, and invests in community educational and outreach programs.

COMPRES NSF Cooperative Agreements



COMPRES

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

COMPRES I: 2002-2007
COMPRES II: 2007-2012
COMPRES III: 2012-2017
COMPRES IV: 2017-2022

Figure 1. COMPRES Cooperative Agreements.

The history of COMPRES started with a Town Hall gathering at the Fall 2000 AGU Meeting in San Francisco organized by the AGU Mineral and Rock Physics Committee, and the planning continued with a workshop at the Scripps Institution of Oceanography in La Jolla in February 2001, and culminated in a successful proposal to the NSF Division of Earth Sciences in August 2001. In May 2002, a Cooperative Agreement was established which funded COMPRES from 2002-2007. This cooperative agreement was renewed in 2007 (COMPRES II), and in 2012 (COMPRES III, the current cooperative agreement). In this proposal we present our vision for the next five years (COMPRES IV), as defined by the science goals that drive our research in mineral physics and adjacent fields, and built on a foundation of our community's recent accomplishments and track record of service.

2.1. COMPRES Administration and Governance

Figure 2. Locations of the COMPRES member institutions in the US (U. Hawaii also a member).

COMPRES is a community based research support organization consisting of educational and not-for-profit US institutions with research and educational programs in mineral physics and/or related fields. An electorate consisting of member institutions (rather than individuals) charts the future and establishes priorities of the consortium via governance processes clearly defined by the COMPRES Bylaws. Other organizations and non-US institutions are eligible to be affiliated members with a non-voting representative. As of July 2016, COMPRES had 68 member institutions (Figure 2), and 50 affiliate institutions overseas. COMPRES has a bicameral organization, with the President acting as PI of the organization, and the member institutions represented by the Executive Committee and two standing committees, the Facilities Committee, and the Committee on Infrastructure Development, Education and Outreach (Figure 3).

The ***President*** of COMPRES is the Chief Executive Officer of the organization and executes all contracts and agreements on behalf of the organization and carries out the directives of the Electorate. In relationship to the NSF Cooperative Agreement, the President of COMPRES also serves as the Principal Investigator. The President is appointed by the Executive Committee, in consultation with the cognizant NSF Program Director, and with a recommendation from an ad hoc Search Committee. Jay Bass (UIUC) served as the first COMPRES President starting in 2002, and he was succeeded by Robert Liebermann (Stony Brook University) who held the post from 2003 to 2010. When Professor Liebermann stepped down, Jay Bass again accepted the President role and served until November 2015. When Professor Bass announced in 2014 his plans to step down, the Executive Committee appointed a Search Committee to lead the effort to find a new President. The Search Committee was chaired by Robert Liebermann with other members Michael Brown (U. Washington), Kanani Lee (Yale U.), Tom Duffy (Princeton U.), Guy Masters (UC San Diego), and William McDonough (U. Maryland). Carl Agee of the University of New Mexico was offered the position of President of COMPRES and he accepted. Professor Agee assumed the role of President on November 15, 2015 and the Central Office of COMPRES and the Cooperative Agreement was transferred to the University of New Mexico. The Central Office staff includes Dr. Agee (President), Shannon Clark (Program Director), and Beth Ha (Administrative Assistant). The Institute of Meteoritics (IOM) at UNM is providing in-kind support to the COMPRES Central Office that is necessary for COMPRES operations. UNM is providing additional office space for the COMPRES Central Office adjoining the IOM Director's office. COMPRES benefits from the IOM administrative structure that was already in place when the Central Office was transferred from UIUC.

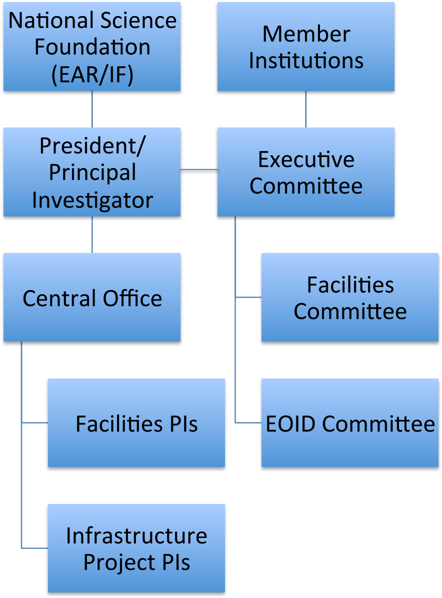


Figure 3. COMPRES organizational chart.

Figure 4. COMPRES Presidents at the Fall 2015 AGU, Town Hall Meeting. Jay Bass (L), Bob Liebermann (C), Carl Agee (R).

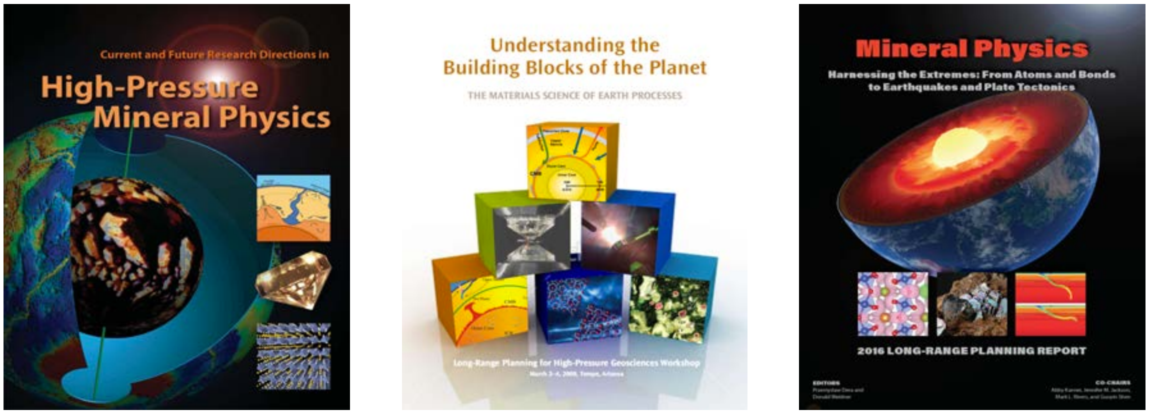
The ***Executive Committee*** plays a central role in representing the COMPRES Electorate by formulating and approving all activities of the organization. Two standing committees, the ***Facilities Committee*** and the ***Education, Outreach and Infrastructure Development Committee (EOID)*** are responsible for evaluating existing COMPRES programs and reviewing new proposals. The Facilities Committee evaluates the effectiveness of the COMPRES user facilities, and helps coordinate among facilities such as different beamlines to maximize benefits to our community. This committee provides ongoing evaluation of the facility needs of the mineral physics community and makes recommendations to the Executive Committee regarding changes in the levels of support of all community facilities, including introduction and sunset of facilities. The EOID Committee reviews infrastructure development and education and outreach projects that are supported by COMPRES. It evaluates proposals by the community for new development projects and makes funding recommendations to the Executive Committee and president. The EOID Committee also assesses how COMPRES education and outreach projects serve community needs and recommends continuation and/or changes. Each year, all COMPRES-funded facilities and EOID projects submit a detailed report of the previous year's activities, and plans and budget request for the upcoming year. These documents are reviewed and evaluated by the standing committees before and during a series of annual AGU breakfast meetings. Comprehensive reviews including occasional site visits of COMPRES and related facilities are an essential part of quality control for the organization. The most recent facility site visit was in Fall 2015 to the NSLS-II facilities.

2.2. Community Input and Planning for this Proposal

The process of community preparation for this proposal began in October 12-14, 2014 with an NSF-funded "Mineral Physics Planning Workshop" held at Argonne National Laboratory. Fifty-six people attended the workshop, which featured plenary talks by Bruce Buffett, Mark Rivers, William McDonough, Kerstin Lehnert, George Srajer, and Craig Manning. Workshop participants were divided into eight groups to discuss the most exciting accomplishments and

Figure 5. Participants of the Long-Range Planning Workshop held in Argonne, Illinois, on October 10–12, 2014.

future opportunities from different perspectives. Four of the groups focused on scientific concepts: (1) equation of state, (2) extreme petrology, (3) transport properties: thermal, electrical, magnetic, and (4) rheology/grain boundary/diffusion. Similar to the two previous long-range planning workshops, the goal of the Argonne workshop was to produce a written long-range vision report, following the examples of the 2003 Bass Report and 2009 Williams Report. This document (aka the Dera-Weidner Report) is the result of these efforts. An editorial team coordinated the writing effort, and included co-editors P. Dera and D. Weidner, as well as co-chairs of the workshop committee, J. Jackson, A. Kavner, M. Rivers, and G. Shen. Online versions of these reports are available at http://compres.us/publications.



“Bass Report” (2004) **“Williams Report” (2009)** **“Dera-Weidner Report” (2016)**

Figure 6. Long-Range Planning Documents, Bass et al. (2004), Williams et al. (2009), and Dera et al. (2016).

The next step in the community planning process was an open discussion of opportunities and long-range research needs held at the June 2015 COMPRES Annual Meeting at Cheyenne Mountain, CO. A COMPRES Town Hall meeting was held in San Francisco during the Fall 2015 AGU meeting in which presentations were given on possible projects for the renewal proposal. Presenters included Jay Bass (UIUC), Tom Duffy (Princeton Univ.), Jie (Jackie) Li (Univ. Michigan), Bob Liebermann (Stony Brook Univ.), Zhenxian Liu (Carnegie Institution), Wendy Panero (Ohio State Univ.), Mark Rivers (Univ. Chicago), Dan Shim (Arizona State Univ.), Quentin Williams (UC Santa Cruz), Dongzhao Zhang, (Univ. Hawaii), and Jin Zhang, (Univ. Hawaii). In early 2016, the COMPRES Executive Committee issued a call for facility and EOID proposals for inclusion in the renewal proposal. The proposals received were evaluated by the Facilities and EOID Committees who made recommendations to the Executive Committee. Final decisions on which projects to include in the renewal and budget levels were made by the Executive Committee in consultation with the President. A community-wide discussion of the status and plans for the renewal proposal was then held at the 2016 COMPRES Annual Meeting at the Tamaya Resort in Santa Ana Pueblo, NM, where the community provided additional input to the Executive Committee and the President on the emerging shape and structure of this proposal.

2.3. Science Motivation and Facility and Infrastructure Needs

The mission of COMPRES is to enable compelling materials research in Earth science by providing world-class facilities and the best infrastructure possible for the high-pressure geosciences community. Our community's instrument and facility requirements for the upcoming five years (June 2017 – May 2022) are therefore driven by the community's best efforts to couple between the most pressing scientific questions and the state of the art technologies that will help us address them. The COMPRES community pursues a broad array of scientific questions related to the physical and chemical workings of the Earth's interior, including:

(1) Determining the mineralogy and composition of the deep Earth by acquiring data on the elastic properties, anisotropy, and equations of state, which are then applied to interpreting seismic models.

(2) Understanding mantle dynamics and plate tectonics, which requires knowledge of the rheological properties of minerals and rocks determined at mantle pressure-temperature conditions.

(3) Elucidating Earth's deep water and carbon cycles, including the sequestration and circulation of water and carbon in the mantle, how it relates to whole-Earth volatile cycles, and their origins during planetary accretion and evolution, which requires quantitative constraints on the effects of water on mineral properties at high pressure.

(4) Revealing the nature of the Earth's lower mantle through petrological investigations, including yet unexplored aspects of multi-component systems, including phase equilibria and melting relations, redox behavior of multivalent species, and storage and distribution of volatiles, and storage and distribution of trace elements and isotopes amongst deep Earth phases.

(5) Shedding light on the nature and dynamics of the core-mantle boundary by investigating the physical and chemical properties of deep lower mantle minerals such as the post-perovskite phase and their interaction with silicate and metallic melt.

(6) Understanding core composition, geodynamo generation, planetary thermal structure, and the evolution of the inner core/outer core system by studying the properties of iron alloys and metallic melts at core conditions.

(7) Exploring exotic geological processes of the inner planets of our solar system, the moons of the giant outer planets, and newly discovered Earth-sized and super-Earth extra-solar planets by measuring material properties at relevant pressure-temperature-and composition space.

In recent years, synchrotron-based techniques have played an ever-increasing role in experimental efforts to measure properties of geological materials at high pressures and temperatures. The new generation of synchrotrons at national facilities offers great promise for the coming five years and beyond (Figure 7) . At Brookhaven National Laboratory, the recently completed $0.9 billion facility NSLS-II will produce X-rays over 10,000 times brighter than the original National Synchrotron Light Source (NSLS) while providing improved spatial and energy resolution as well as a world-leading synchrotron-infrared (IR) radiation source. At the same time, plans are underway to provide a major upgrade to the Advanced Photon Source (APS) at Argonne National Laboratory. Upgrades are also planned for the Advanced Light Source (ALS) synchrotron at Lawrence Berkeley National Laboratory. These new or upgraded synchrotrons augmented by improvements in x-ray optics and detectors will lead to dramatic advances in measurements at high pressure.

Figure 7. The Department of Energy (DOE) –supported synchrotron light sources where COMPRES provides support for user facilities.

Thus the upcoming period spanned by COMPES IV, and beyond, offers exciting opportunities as new and upgraded synchrotron facilities will dramatically improve experimental capabilities and allow for novel experiments needed to test hypotheses and address yet-unanswered questions on the nature of deep planetary interiors. Infrastructure and facility developments supported by COMPRES are at the same time creating a new generation of tools and techniques for attaining and measuring the deep interior environment in the laboratory.

Throughout the process of evaluating ongoing facilities and new projects for this proposal, the COMPRES Executive Committee has developed four broadly defined working guidelines (aka Abby's rules). These evaluation criteria are:

1. ***Science***: What are the scientific drivers motivating the facility/infrastructure investment? What are the science outcomes from previous investment?
2. ***User Community***: Who are the users of the facility? Do COMPRES investments provide a resource that is open for the community? Is the user group inclusive, representing a diversity of practitioners, and with opportunities for new users?
3. ***Management Team***: What is the nature of the partnership between COMPRES and the PI(s)? Are COMPRES beamline scientists provided with high-quality support, supervision, mentoring, and professional development?
4. ***Facility:*** How does the facility help match the needs of the COMPRES community? Does the facility provide state of the art equipment? What is the nature of the partnership between COMPRES and the host facility, both on paper (e.g. MOUs/Partner User Agreements) and in practice? To what extent does COMPRES leverage additional support from the host facility (e.g. equipment, shared facilities, and/or staffing)?

2.4. Summary of Existing COMPRES-Supported Facilities

Below are brief summaries of the facilities currently managed by COMPRES. In this renewal proposal, the most significant new request is to add an innovative high tonnage large volume press user facility to the COMPRES portfolio, and we outline some options designed to help foster a world-class facility in the United States, analogous to existing facilities in Europe (Bayreuth), Japan (Matsuyama) and underway in China. Additionally, we propose enhancing the level of support at selected existing facilities, in particular the Frontier Infrared Spectroscopy (FIS) IR beamline diamond anvil cell (DAC) at NSLS-II that will start accommodating COMPRES users for its offline system this year, and online when the beamline becomes operational in 2018. Finally, we anticipate sun-setting facilities that have served their short-term purpose, for example facilities that helped the community negotiate the "dark period" transition between NSLS and NSLS-II. An extended summary of each of the proposed projects going forward into the COMPRES renewal is detailed in the Research Plan (cf. section 4). Additional information about each of the proposed facilities (including new and continuing facilities) is provided in Appendix A.

ALS 12.2.2 Beamline (PI: Quentin Williams, UC Santa Cruz)

This beamline enables monochromatic X-ray diffraction experiments at high pressures and temperatures using the diamond anvil cell. The beamline specializes in studies of mechanical strength, deformation and texture development at ultra-high pressures using the radial diffraction technique in the diamond cell; development of innovative high temperature methods including laser heating and internal resistance-heating; and a newly-developed program in single-crystal X-ray diffraction. The facility has a new, completely redesigned temperature measurement system allowing measurements of thermoelastic equations of state and pressure-temperature phase stability.

APS Beamline 13BM-C PX^2 (PI: Przemek Dera, Univ. Hawaii)

The Partnership for eXtreme Xtallography (PX^2) facility is a collaboration between COMPRES and GSECARS. *(Note: GSECARS is an independent user facility based at the APS covering a broad array of synchrotron-based geoscience studies, and run out of the University of Chicago).* The 13BM-C end station provides high energy monochromatic X-rays for a variety of diffraction techniques to examine mineral structures at high pressures in the diamond anvil cell. This beamline is optimized for a variety of advanced crystallography experiments including powder and single crystal structure determination, equation of state studies, and thermal diffuse scattering. PX^2 opened for general users in APS in 2015.

APS Gas Loading for DAC (PI: Mark Rivers, Univ. Chicago)

The Gas Loading System is the second collaboration between COMPRES and GSECARS, and provides both an in-house and a mail-in service to load diamond anvil cells with a variety of hydrostatic gas pressure media such as neon or helium. This has been a major advance for the U.S. diamond anvil cell community, improving the quality of data collection, and setting the standard for mineral structure, phase stability, and equation of state measurements at high-pressures. COMPRES supports the open "mail-in" service, used by scores of diamond cell researchers regardless of whether they are performing experiments at GSECARS, at another APS sector, at another synchrotron, or in their home laboratory.

APS Inelastic X-Ray Scattering Facilities at Sector 3 (PI: Jay Bass, UIUC)

COMPRES has supported and helped develop a series of synchrotron-based spectroscopy techniques harnessing the inelastic x-ray scattering behavior of ^{57}Fe at extreme temperatures and pressures. This suite of techniques, which includes Nuclear Resonant Inelastic X-ray scatting (NRIXS) and High Energy Resolution Inelastic X-Ray Spectrometry (HERIX-3), can be used to probe sound velocities, spin state, site occupancy, and vibrational properties of iron in metals and oxides relevant to the deep Earth. In addition, an offline (non-synchrotron-based) Mössbauer Spectroscopy system is housed at the APS and managed by PI Bass, in collaboration with Ercan Alp (Univ. Chicago).

Multi-Anvil Development/Cell Assembly Program (PI: Kurt Leinenweber, ASU)

This longstanding program provides standardized cell parts for multi-anvil experiments and develops new types of cells for novel experiments. The supply of routinely-used multianvil cell assemblies to a large community of multi-anvil press laboratories around the world is a self-supporting service via a fee structure. COMPRES provides additional support to the ASU team to improve MAP technology through design of novel assemblies for new types of experiments.

NSLS-II Beamline Frontier Infrared Spectroscopy (FIS) Facility for Studies under Extreme Conditions (PIs: Russell Hemley, Zhenxian Liu, GWU)

The Frontier Synchrotron Infrared Spectroscopy (FIS) Beamline will replace and enhance the COMPRES IR operations previously installed at the NSLS beamline U2A. The infrared beamline is among the high-priority "NextGen" beamlines already funded and currently under construction at the NSLS-II. Two current COMPRES Executive Committee members (Panero and Jacobsen) are members of the DOE Beamline Advisory Team (BAT) overseeing the design and construction of FIS. The beamline is scheduled to deliver beam in early 2018, and will provide a facility dedicated to high-pressure infrared (IR) measurements using the diamond-anvil cell, and offer the flux and spatial resolution necessary to study volatiles in challenging samples from nature requiring micrometer spatial resolution. To bridge the gap between NSLS and NSLS-II, an offline infrared lab including FTIR and Raman spectroscopic systems has been established and housed at the NSLS-II and is available for users during the transition period. Capabilities include studies of the presence and structural state of water in minerals, thermodynamic properties, electronic structure, and thermal conductivity at extreme conditions.

NSLS-II XPD and APS 6BM-B Beamline Multi-anvil (PI: Donald Weidner, Stony Brook)
The project at the X-ray powder diffraction (XPD) beamline is dedicated to multi-anvil press (MAP) research and will replace COMPRES MAP operations previously at NSLS beamline X17, but with monochromatic beam allowing angle dispersive x-ray diffraction and imaging capabilities. The XPD beamline is one of the First Generation beamlines that has been built at NSLS-II, and we estimate a start of the full user operation of the press in November 2016. Research will be in the areas of rheology and ultrasonic sound velocity measurements at high pressures and temperatures. During the current dark period, equipment from the shuttered NSLS, together with additional Stony Brook equipment was configured and installed at the APS beamline 6BM-B to provide an interim high pressure facility with some of the capability that existed at the X17-B2 beamline. Now that the installation of the XPD multi-anvil facility at NSLS-II is nearly complete, we plan to decommission the APS beamline 6BM-B multi-anvil project and redirect funds at the end of year-1 in COMPRES IV (June 1, 2018). This project will have served the COMPRES multi-anvil community satisfactorily as an interim facility during its three-years of activity (2015-2018).

Note about user access to COMPRES supported synchrotron facilities
Normally COMPRES users' requests for beamtime are submitted through a general user proposal system at each synchrotron. Proposals are evaluated on scientific merit and feasibility by a review panel and beamtime is assigned to the highest-ranking proposals, regardless of discipline or COMPRES-affiliation. All of the COMPRES synchrotron facilities are in high demand and typically oversubscribed by a factor of two (defined as the ratio of beamtime shifts requested to those available and allocated). Thus, most beamtime requests at these facilities receive less time than requested and many are declined.

COMPRES- supported beamlines, because of our significant investment in personnel and equipment, have special relationships with their host synchrotron managements through written memoranda of understanding, such as Partner User Agreements (PUA). A PUA can guarantee additional beamtime for COMPRES users through various mechanisms, depending on the way user time versus partner time is allocated at the facility. This special access enables COMPRES users to get additional time on our beamlines in over and above the allocations awarded through the general user proposal review system. These agreements are negotiated between the President of COMPRES and the cognizant facility representative, and are vetted and voted on by the Executive Committee.

2.5. New Facilities proposed for COMPRES IV

Large Volume Multi-anvil Press Facility
Many high-priority science questions of the COMPRES community require large samples of deep Earth phases synthesized at high pressures with reliable control of complex sample environment. Japanese and European scientists have made strides in developing multi-anvil press technologies for sample synthesis for community members to enable unique high pressure measurements. Our goal is to combine a large volume press user facility for sample synthesis with community access to state-of-the-art post-mortem analysis techniques such as electron microscopy and chemical microprobe. COMPRES is excited about helping to spearhead a prospective large volume multi-anvil press user facility in the United States. We envisage that COMPRES would support such a facility by providing partial funding for salaries, materials and supplies. At present we have two high quality proposal options that are under consideration. Both projects are seeking equipment funding for the large press through sources external to COMPRES. The CETUS project is seeking funds from the NASA Planetary Major Equipment Program (PME) and the LMAP project is planning on competing for an NSF Major Research Instrumentation (MRI) grant. The outcome of these equipment proposals will not be known until late 2016 or early 2017. If they are successful in securing the equipment awards and one of them is selected by COMPRES as a new facility, then support for the project would appear in the COMPRES budget no earlier than year-2 (2018). Here is a brief summary of both proposals; 2-page descriptions can be found in section 4 “Research Plan”, and more detailed descriptions are in Appendix A.

Community Extreme Tonnage User Service (CETUS): A 5000 Ton Press Open Research Facility in the United States (PI: Lisa Danielson, Jacobs Technology, NASA Johnson Space Center)
Community Extreme Tonnage User Facility (CETUS) is a proposal to establish a large sample volume, 5000 ton multi-anvil press, at NASA Johnson Space Center in Houston, TX. CETUS would be an open user facility for COMPRES members and the entire research community, with the unique capability of a 5000 ton press, supported by a host of existing co-located experimental and analytical laboratories and research staff. This staff consists of 19 researchers and technicians and includes three broad areas of expertise: experimental petrology, microbeam analysis, and geochemistry.

Community Large Multi-Anvil Press Facility (LMAPF) at ASU through COMPRES (PI: Kurt Leinenweber, ASU)
LMAPF is a project to build a National Multi-Anvil Facility by combining a new 6000-ton press together with existing two 1000-ton presses, two 300-ton presses, and other high-pressure equipment at Arizona State University (ASU). The facility would serve the COMPRES community by 1) supporting users in conducting experiments and synthesis of large quantity, high-quality samples at deep mantle pressure-temperature conditions in a large multi-anvil press (LMAP) and 2) developing and distributing new materials for anvils, gaskets, and heaters, which will enable existing high-pressure devices (including piston cylinder, multi-anvil presses and diamond-anvil cells) in the US to achieve much higher pressure-temperature conditions than are currently achievable.

2.6 Education, Outreach, Infrastructure, and Development (EOID)

COMPRES's EOID program is our investment in the future—charged with technological development and cultivation of human resources. Our ID program fosters the development of new methods and approaches for high-pressure research, shepherding new capabilities at synchrotrons and ensuring that offline user facilities are accessible to the entire COMPRES community. By combining infrastructure development with education and outreach, COMPRES can provide broad and cost-effective training to inform our community of emerging opportunities, and to help our existing research be more efficient, productive, and accurate. This program is structured to be a nimble responder to COMPRES community initiatives and needs. When COMPRES invests in a new technology or facility, the consortium follows through with education and outreach to inform the community and to build the user base. In some cases, EOID projects evolve into long-term activities that can become COMPRES facilities. Examples of EOID projects maturing into sustainable facilities are the Multi-anvil Project at ASU, the APS Gas Loading Facility, and the APS IXS Facility at Sector 3. In other cases, the scope of the EOID project might be shorter term–for example either a one-time workshop, or a project designed to be supported for 1-3 years and then spun-off to other support structures (or shared support). We propose to support three new EOID projects in the first two years of COMPRES IV and these are briefly listed here, with more details in section 5 "Research Plan", and full project descriptions in Appendix A of this proposal.

A Career Path for African-American Students from HBCUs to National Laboratories (PI: Robert Liebermann, Stony Brook)
Research organizations such as COMPRES need to specifically invest in cultivating diversity in our scientific workforce. We have an opportunity to build on an existing program seeded at Stony Brook to provide pathways specifically targeted towards African American students to train in mineral physics. COMPRES proposes a new initiative to recruit undergraduate science and engineering students from underrepresented groups that received their undergraduate degrees from minority-serving institutions to pursue a Master's degree in the Department of Geosciences at Stony Brook University, and upon graduation, be qualified for a variety of career tracks, including employment at DOE-sponsored national user facilities, continuation for more graduate study in a geophysics PhD program, and/or other employment paths that are enhanced by a hands-on technical training.

Infrastructural Development for Deep-Earth Large-Volume Experimentation "DELVE" -- An EOID initiative for doubling pressure capability of large-volume apparatus in the U.S. (Team members: Yanbin Wang, Tony Yu (GSECARS, APS), Donald Weidner, Matthew L. Whitaker, Baosheng Li (SBU, NSLS-II), Kurt Leinenweber (ASU), Zhicheng Jing (CWRU), Bin Chen (U Hawaii, Manoa), Yingwei Fei (CIW), Charles Lesher (UC Davis), Shun-ichiro Karato (Yale)
Whereas the CETUS and LMAPF projects described above are focused on sample synthesis for general users and aimed at increasing sample volume in the US at pressures extending down to the uppermost lower mantle (~30 GPa), this two-year project supports development of techniques in multi-anvil presses of member laboratories in the US and to double the current pressure capability to ~50 GPa with a wide range of in-situ techniques. Eleven team members from nine institutions will work together in this initiative, with three scientific goals in mind: mineral elasticity, melt properties, and lower-mantle petrology. The results from this project will

be used to jump start larger scientific initiatives such as grand challenge proposals to the NSF aiming at addressing deep-earth geophysical problems.

Development of an Electrical Cell in the Multi-Anvil to Study Planetary Deep Interiors (PI: Anne Pommier, UC San Diego)
This project is to design and test a conductivity cell that allows advances in electrical measurements at pressures > 8 GPa using the multi-anvil apparatus. The objective of this project is two-fold: 1) technically, to develop a standardized electrical conductivity cell that can then be widely used at other high-pressure laboratories and 2) scientifically, as part of the testing of the cell, to measure the conductivity of outer core analogues of two terrestrial bodies, Mercury and Mars.

Other Activities of EOID
In addition to the above projects, each year COMPRES supports a number of workshops, and sponsors a Distinguished Lecturer Series that sends prominent members of our community to colleges and universities to deliver colloquia and to meet with students. Details on the facilities, infrastructure development projects, and education & outreach projects that we propose to continue in a next phase of COMPRES are described in the project plan later in this section. The community aspects of COMPRES cannot be overemphasized. The community defines all the activities of COMPRES and its future directions. Each year there is a call for new EOID proposals to the entire COMPRES community. Any group or individual can propose a technology, service, or community activity for development by COMPRES. Two committees evaluate all proposals before deciding whether to fund or decline. Often workshops will be held to gauge the level of support for a particular technology or activity. Once COMPRES commits to a facility or technology, workshops are often held to inform the community and/or for training. Through this process we build user groups for the best experimental tools available and advance our science. For example, we plan on supporting a user workshop in the spring of 2018 to coincide with the opening of the Frontier Infrared Spectrometry (FIS) facility at NSLS-II. This will inform our community about the opportunities at the new world-leading DAC IR spectroscopy lab. Growing our user base at this new facility is of crucial importance to the success of COMPRES at the NSLS-II. We anticipate that the workshop will encourage new users and existing users of the IR facility at the NSLS U2A to submit General User proposals and make FIS a thriving lab for research on the Earth's deep volatile cycles.

3. Accomplishments: Results of Prior NSF Support 2012-2016

Publications
Through its network of support for US mineral physicists, COMPRES has enabled major advances in experimental studies of the physical and chemical properties of Earth minerals and other relevant materials; COMPRES facilities and infrastructure projects have contributed to 622 peer-reviewed publications and Ph.D. theses during the period 2012-2016. Table 1 shows the number of publications for each facility by year. These numbers are one indication of the strong demand for COMPRES facilities, their productivity, and the broad use of the services COMPRES provides through its facilities. The total publications during COMPRES III is about 5% higher than the previous cooperative agreement period 2007-2011 (COMPRES II had 594

publications). Table 1 shows that many publications are from the operations at NSLS, and even though it closed in 2014, we are still seeing its published results appearing in 2015 and 2016. Although we have yet no entries for NSLS-II XPD and FIS, we anticipate that these new facilities will be productive over the course of COMPRES IV. The recently established Partnership for eXtreme Xtallography (PX^2) facility at APS is already starting to see new publications in 2016; the 6BM-B, dark-period multi-anvil facility at APS has some publications in review at this time.

Table 1. COMPRES Facilities Publication Statistics (2012-2016)

	2012	2013	2014	2015	2016*	Total
12.2.2 DAC ALS	32	31	22	20	11	116
U2A IR DAC NSLS	16	12	20	11	10	69
X17B2 Multi-anvil NSLS	12	13	14	5	6	50
X17C/B3 DAC NSLS	34	34	51	27	20	166
Sector 3 IXS DAC + Mössbauer APS	10	3	13	9	6	41
Gas Loading APS	17	23	24	40	24	128
ASU Multi-anvil	3	9	9	7	3	31
PX^2 APS	N/A	N/A	N/A	N/A	3	3
6BM-B Multi-anvil APS	N/A	N/A	N/A	N/A	0	0
EOID other	4	4	0	0	0	8
Central Office	2	8	0	0	0	10
Total	130	137	153	119	83	622

Peer-reviewed publications and theses.

* Through July 2016

Research Highlights

As examples of the breadth and vitality of the scientific achievements of the COMPRES community, Appendix B of this proposal provides 143 research "one-pagers" that have been submitted by research groups in preparation of this proposal. These "one-pagers" summarize selected published results enabled by COMPPRES investment in community facilities and infrastructure from 2012-2016, and are roughly organized by scientific theme (though many of the contributions crosscut more than one theme).

In this section, we present a selection of contributions highlighting exemplary science contributions arising from COMPRES-funded facilities and showing exciting discoveries across a broad range of scientific themes, including temperature and light element composition of the Earth's core, nature of large low-shear-velocity province (LLSVP) in the lower mantle, origin of long-lived geochemical reservoirs in deep Earth, and magmatic processes, global water and carbon cycles, and evolution of atmospheric oxygen.

Dehydration Melting at the Top of the Lower Mantle: In a Letter to *Science*, Schmandt et al., (2014) examined the potential for regional-scale hydration of the lower mantle by subducting slabs. Their study combined laboratory experiments and seismic observations to detect dehydration melting below 660 km in regions of down-welling beneath North America. The research involved a partnership between GSECARS/APS, COMPRES U2A/NSLS, and the NSF-

Earthscope, USArray. In lab experiments, hydrated ringwoodite was used to synthesize the bridgmanite plus ferropericlase assemblage, which routinely produced dehydration melt. In the seismic study, generation of over 100,000 receiver functions recorded by 2,244 stations created a common conversion point (CCP) image of P-to-S converted phases in the depth range of 500-900 km. Negative amplitude Ps conversions (indicative of negative velocity gradients) were observed in regions where geodynamic models show down-welling across the 660-km discontinuity, a likely indicator of widespread hydration of the mantle transition zone.

Dry (Mg,Fe)SiO$_3$ Perovskite in the Earth's Lower Mantle: In a *JGR* article, Panero et al. (2015) combined synthesis experiments and first-principles calculations to show that MgSiO$_3$-perovskite with minor Al or Fe does not incorporate significant OH under lower mantle conditions. The experiments with perovskite, stishovite, and residual melt were synthesized in the diamond anvil cell at 1600-2000 K and 25-65 GPa. Combined Fourier transform infrared (FTIR) spectroscopy (obtained using NSLS IR beamline), x-ray diffraction (performed at NSLS X-17C), and ex-situ transmission electron microscopy (TEM) analysis suggest a maximum water content for MgSiO$_3$-perovskite of 220 ppm H$_2$O, and likely no more than 10 ppm H$_2$O. Complementary, Fe-free, first-principles calculations predict that MgSiO$_3$-perovskite can only dissolve a maximum of 37 ppm H$_2$O at the top of the lower mantle, decreasing to 31 ppm H$_2$O at 125 GPa and 3000 K, in the absence of a melt or fluid phase. They propose that these results resolve a long-standing debate of the perovskite melting curve and explain the order of magnitude increase in viscosity from upper to lower mantle.

Evidence for H$_2$O-bearing Fluids in the Lower Mantle from Diamond Inclusion: In an article published in *Lithos*, Palot et al. (2016) report the first direct evidence for water-bearing melt/fluid in the lower mantle from a natural ferropericlase crystal contained within a diamond from São Luíz, Brazil. Magnesioferrite exsolution occurs topotaxially in the ferropericlase, but also along pre-existing dislocations near the inclusion surface, indicating that exsolution occurred subsequent to ascent and applied stress on the inclusion surface from the diamond cavity. The presence of precipitated brucite–Mg(OH)$_2$ in the ferropericlase crystal reflects the later-stage quenching of an H$_2$O-bearing fluid film previously trapped between the inclusion and diamond host cavity. Quenching of the fluid, likely at transition zone conditions or shallower, resulted in the nano-precipitates of brucite (observed using NSLS-IR beamline U2A). Dehydration melting may be an important process to transport water across the 660-km boundary and generate volatile-rich fluids and a diamond factory near the top of the lower mantle.

Tetrahedrally Coordinated Carbonates in Earth's Lower Mantle: In a *Nature Communications* article, Boulard et al. (2015) reported a new type of bonding in carbonates, which are the main species that bring carbon deep into our planet through subduction. Normally carbonates are fundamentally distinct from silicates in the Earth's crust in that carbon binds to three oxygen atoms, while silicon is bonded to four oxygens. To examine the evolution of the C-O bond at Earth's mantle pressures, the vibrational properties of (Mg,Fe)CO$_3$ was examined using infrared spectroscopy at NSLS U2A beamline as a function of pressure up to 103 GPa. An unequivocal spectroscopic signature of the high pressure phase of (Mg,Fe)CO$_3$ were observed above 80 GPa. Aided by ab-initio calculations, the new high-pressure infrared signature was assigned to C–O bands associated with tetrahedrally coordinated carbon. Tetrahedrally coordinated carbonates are expected to exhibit substantially different reactivity than low-pressure threefold coordinated carbonates, as well as different chemical properties in the liquid state which may have significant implications for carbon reservoirs and fluxes, and the global geodynamic carbon cycle.

Hidden Carbon in Earth's Inner Core Revealed by Shear Softening in Dense Fe_7C_3: In a paper published in *PNAS*, Chen et al. (2014) presented results on iron carbide Fe_7C_3 which has recently emerged as a candidate component of the inner core because at core pressures it is likely the first phase to solidify from a liquid containing iron and a small amount of carbon. In their study, they employed diamond-anvil cell techniques in combination with Nuclear Resonant Inelastic X-ray Scattering, Synchrotron Mössbauer Spectroscopy, and X-ray Emission Spectroscopy, in order to probe the acoustic sound velocities and magnetic transitions of Fe_7C_3 under high pressures. They found exceptionally low shear-wave velocity in the low-spin and non-magnetic phase of iron carbide Fe_7C_3 at high pressures. The anomalously low shear wave speeds observed for the Earth's inner core could be explained by low-spin Fe_7C_3, thus eliminating the need to invoke partial melting or a postulated large temperature dependence of iron shear velocities.

Equations of State in the Fe-FeSi System at High Pressures and Temperatures: In a *JGR* article, Fischer et al. (2014) report measurements of the phase stability and equations of state for stoichiometric FeSi and a Fe-Si alloy using synchrotron-based X-ray diffraction in conjunction with the laser-heated diamond anvil cell. They also calculated equations of state of Fe, $Fe_{11}Si$, Fe_5Si, Fe_3Si, and FeSi using ab initio methods, finding that iron and silicon atoms have similar volumes at high pressures. By comparing their experimentally determined equations of state to the observed core density deficit, they found that the maximum amount of silicon in the outer core is ~11 wt %, while the maximum amount in the inner core is 6–8 wt %, for a purely Fe-Si-Ni core. The good matches between measured equation of state values to the observed density, bulk modulus, and sound speed as a function of depth in the core, suggests that silicon is a viable candidate for the core's dominant light element. (Portions of this study used gas loading system.)

Synthesis of FeO_2 and the Fe-O-H System in the Deep Earth: In a Letter to *Nature*, Hu et al. (2016) explored chemical reactions in the system of Fe-O-H at high-pressure and high-temperature that mimic deep lower mantle conditions. They reported the stability of a new FeO_2 phase that forms when hematite (Fe_2O_3) is compressed in O_2 at or above 76 GPa and 1800 K. The new phase was identified through synchrotron X-ray diffraction at the PX^2 beamline at the APS. The newly discovered FeO_2 phase holds an excess of oxygen and has the same atomic structure as FeS_2. They also observed that the mineral goethite (FeOOH) decomposes to FeO_2 at 92 GPa and 2050 K by releasing hydrogen. It is hypothesized that in the deep lower mantle environment, the hydrogen released from FeOOH would diffuse, infiltrate or react to form hydrocarbons or other volatiles, while FeO_2 products would remain in the mantle and accumulate over time through delivery by plate tectonics.

Redox-induced Density Contrast and Implications for Mantle Structure and Primitive Oxygen: In a *Nature Geoscience* article, Lee et al. (2016) presented experiments in which they subjected two synthetic samples of nearly identical composition that are representative of the lower mantle (enstatite chondrite), but synthesized under different oxygen fugacities, to pressures and temperatures up to 90 GPa and 2,400 K. Compression of the more reduced material produced Al_2O_3 as a separate phase as well as the abundant mineral of the lower mantle, bridgmanite, and the resulting assemblage is about 1-1.5% denser than in experiments with the more oxidized material. Their geodynamic simulations suggest that such a density difference can cause a rapid ascent and accumulation of oxidized material in the upper mantle, with descent of the denser reduced material to the core–mantle boundary. The resulting heterogeneous redox conditions in Earth's interior can contribute to the large low-shear velocity provinces in the lower mantle and the evolution of atmospheric oxygen. (Project used gas-loading system.)

Temperature of Earth's Core Constrained from Melting of Fe and $Fe_{0.9}Ni_{0.1}$ at High Pressures: In an *Earth and Planetary Science Letters* article, Zhang et al. (2016) reported measurements of the pressure-dependent melting of fcc- and hcp-structured $Fe_{0.9}Ni_{0.1}$ and Fe up to 125 GPa using laser heated diamond anvil cells, synchrotron Mössbauer spectroscopy, and a recently developed fast temperature readout spectrometer. They were able to extrapolate a melting of the $Fe_{0.9}Ni_{0.1}$ composition, providing an upper bound estimate of the temperature of Earth's inner core–outer core boundary, thus providing an estimate for the temperature at the top of the outer core, and the temperature gradient across the core-mantle boundary.

Equation of State of Pyrite to 80 GPa and 2400 K: In a paper published in *American Mineralogist*, Thompson et al. (2016) reported X-ray diffraction measurements on FeS_2 at 15 to 80 GPa and temperatures up to 2400 K using laser-heated diamond anvil cells. Combining their new P-V-T data with previously published room temperature compression and thermochemical data they derived a thermoelastic equation of state for pyrite. When compared with observations of wave speeds in the Earth's core, the results suggest that sulfur alone cannot satisfy both the observed density and wave velocities, and hence the sulfur content needed to satisfy density constraints using the new FeS_2 equation of state should be considered an upper bound for sulfur in the Earth's core. (Project used the COMPRES/GSECARS gas-loading system at APS.)

Slab Stagnation in the Shallow Lower Mantle Linked to an Increase in Mantle Viscosity: In a *Nature Geoscience* article, Marquandt and Miyagi (2015) reported angle-dispersive high-pressure radial x-ray diffraction (rXRD) on $(Mg_{0.8}Fe_{0.2})O$ and $(Mg_{0.9}Fe_{0.1})O$ powder obtained at beamline 12.2.2 of the Advanced Light Source (ALS). Deformation of ferropericlase was accomplished in a panoramic diamond-anvil cell to a maximum pressure of 100 GPa using x-ray transparent cubic boron nitride (cBN) and beryllium gaskets. They observed an initial increase of all lattice strains, followed by saturation by 20 GPa. Starting from ~20-30 GPa they observed a pronounced increase of lattice strains corresponding to (111), (220) and (311) lattice planes. This secondary increase in observed lattice strains continued to ~65 GPa at which point the lattice no longer showed signs of additional elastic strain. From this data, they calculated that the flow strength of ferropericlase increases by a factor of three at pressures from 20 to 65 GPa. Modeling shows that mantle viscosity near subducting slabs could increase significantly by depths of ~900 km, potentially leading to slab stagnation in the mid lower mantle.

Shear Deformation Experiment of Bridgmanite + Magnesiowüstite Aggregates Under Lower Mantle Conditions: In a Report to *Science*, Girard et al. (2016) showed the results of large strain deformation experiments on a lower mantle assemblage using a rotational Drickamer apparatus at beamline X17B at the NSLS. They found that bridgmanite is substantially stronger than magnesiowüstite and that magnesiowüstite largely accommodates the large strains generated in the experiment. The results suggest that strain weakening and resultant shear localization likely occur in the lower mantle. This would explain the preservation of long-lived geochemical reservoirs and the lack of seismic anisotropy in the majority of the convecting lower mantle.

Ultralow Viscosity of Carbonate Melts at High Pressures: In a *Nature Communications* article, Kono et al. (2014) reported viscosity measurements on carbonate melts up to 6.2 GPa using a newly developed technique of ultrafast synchrotron X-ray imaging in the Paris-Edinburgh press at the APS. These carbonate melts, which are dominated by the CO_3 trigonal planar units, display ultralow viscosities in the range of 0.006-0.010 Pa s, which are ~2 to 3 orders of magnitude lower than those of basaltic melts in the upper mantle. As a result, the mobility of carbonate melts (defined as the ratio of melt-solid density contrast to melt viscosity) is ~2 to 3 orders of magnitude higher than that of basaltic melts. Such high mobility has significant influence on magmatic processes involving carbonatites. (COMPRES provided seed equipment support to help establish the Paris Edinburgh press at Sector 16 at APS.)

4. Research Plan

Since its inception 15 years ago, COMPRES has leveraged access to national synchrotron facilities (Figure 7) to develop and support a broad array of advanced technologies for high-pressure experimentation. This has broadened the mineral physics user community, forged new developments enabling state-of-the-art measurements of the physical and chemical properties of Earth materials, and ultimately has helped provide new insights into the current state and evolution of Earth and planets.

This section details COMPRES's request for continuing and new research facilities. These facility descriptions show our commitment to maintaining, upgrading, and continually enhancing our current facilities while targeting the development of new innovative technologies to expand the range and improve the quality of experiments, and to enhance our community's contribution to our understanding of the nature and dynamics of the Earth and planets. Here we outline the capabilities that COMPRES provides to our community, our vision for developing these capabilities and ensuring appropriate support and access for high-pressure experimental needs at these existing facilities, and our plan for taking advantage of the opportunities at new and upgraded facilities over the next five years. We are especially excited about our community's presence on the ground floor at the newly established next generation synchrotron light source, the NSLS-II.

4.1 COMPRES Research Facilities

4.1.1. ALS 12.2.2 Beamline DAC (PI: Quentin Williams, UC Santa Cruz)

We request funding to augment and continue COMPRES-associated experimental support and capabilities at Beamline 12.2.2 of the Advanced Light Source.

Our overarching scientific goals are to ensure that our facility has the capabilities to: (1) measure the detailed single-crystal structures of materials at conditions that span those present within subduction zones (using externally-heated single-crystal diffraction); (2) measure polycrystalline X-ray diffraction of materials in axial geometries *in situ* at pressures and temperatures spanning those within Earth's mantle and into Earth's outer core (using our on-line laser-heating system); (3) characterize the strengths of Earth materials at a broad suite of mantle conditions (by deploying our radial diffraction capabilities); and (4) conduct complementary measurements on samples prepared or synthesized at our beamline using techniques around the ALS ring, including Laue microdiffraction and high-pressure infrared spectroscopy.

The first of these goals ties directly in with the widespread interest among much of our COMPRES user community in how volatiles cycle between the crust and interior of the planet, with relevance to topics as diverse as the abundance of water and CO_2 within our planet, the genesis of partial melts at depth in Earth's mantle, and the effects on rheology of silicates within the Earth. We anticipate that single-crystal results at simultaneous high pressures and temperatures will not only show how volatiles are bound into minerals at high pressures and temperatures, but also prospectively demonstrate how they are released from minerals under extreme conditions. The second goal is oriented around characterizing among the most difficult-to-measure and hence ill-constrained parameters at conditions representative of those of the deep planet: the viscosity of deep Earth constituents. This is an area of intense geodynamic and geophysical interest, with experiments that are notorious for their complexity and difficulties: our primary goals here are to not only facilitate producing more homogeneous sample thermal environments, but also to make such strength measurements substantially more straightforward for the non-expert user.

Finally, our last two goals are oriented towards producing more comprehensive characterizations of samples that are synthesized at 12.2.2. Infrared spectroscopic data can illuminate local bonding environments within samples held *in situ* at pressure (for infrared spectroscopic data), which is highly complementary to X-ray diffraction data. In turn, microdiffraction data can constrain phase assemblages (and their distribution) within quenched samples, as well as chemical variations when these generate shifts in microdiffraction patterns: such data can provide key textural and chemical information on the complex assemblages generated within the laser-heated diamond cell.

Most of the budget requested for this project is for personnel: these are currently Drs. Christine Beavers and Jinyuan Yan. Modest additional funding is requested for expendable supplies for COMPRES users, and for travel to major meetings and to interact with other facilities. COMPRES funding has heavily leveraged both additional personnel support and equipment funding from the Advanced Light Source/DOE: two additional personnel are primarily associated with the beamline from the ALS side (Dr. Martin Kunz and Andrew Doran), and the ALS has funded both a high-pressure gas-loading apparatus and a state-of-the-art single-crystal diffractometer and detector over the course of COMPRES-III, which are each high-priced items of primary interest to COMPRES users. The roles of the funded personnel are: (1) to provide assistance to COMPRES users of the beamline; (2) to develop and set-up new techniques and software to benefit the user community; and (3) to conduct their own research (which is often collaborative), which maintains their engagement with state-of-the-art advances in the field. With respect to user support, it is important to note that over the past five years, the available user time at 12.2.2 that has been assigned to COMPRES-affiliated users has varied between 55% and 75% of that available within each 6-month user cycle.

The ALS COMPRES enterprise mandates 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 plan to ensure that the substantial numbers of users with these needs continue to receive high-level support. To enhance user experience, we plan to improve the level of automation of pressure and temperature control, as well as provide sample spatial mapping as part of our routine beamline operations. We also have outstanding sample preparation facilities for high-pressure experiments that include a state-of-the-art laser milling set-up for micro-machining, a gas-loading apparatus, and a redesigned sample preparation laboratory.

Alongside the ALS COMPRES commitment to providing user-friendly support for relatively “routine” experiments, during COMPRES IV we will focus our technical developmental work on several areas of broad interest within the high-pressure geosciences community. First, we will continue development and optimization of our capability for simultaneous high-pressure and high-temperature single-crystal x-ray diffraction studies in the diamond cell. In particular we will couple stable and high-temperature external heating with our new diffractometer system. Second, we will expand and augment our radial diffraction capabilities, particularly on high-temperature experiments with low-thermal gradients, which couple external heating with our recently improved laser-heating system. This work will likely extend our successes during COMPRES-III in expanding the limits of external-heating measurements in the diamond cell. Third, we expect to enhance our interactions with the highly complementary 12.3.2 microdiffraction beamline, which can characterize the structural and chemical differences in quenched high-pressure 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. Fourth, we plan to continue to synergistically work with the very recently redeveloped (by COMPRES) high-pressure infrared spectroscopic capabilities at ALS beamline 1.4.4. Here, the prospect exists to establish a “one stop” shop at the ALS for both high-pressure synchrotron diffraction and infrared spectroscopic measurements in the diamond cell. An important caveat to these areas of focus is that there is a strong possibility that over the next several years our detailed directions might shift in response to user interests or unanticipated advances: we believe that reactively responding to serendipitous developments is a major feature of this facility’s past successes.

4.1.2. APS Beamline 13BM-C PX^2 DAC (PI: Przemek Dera, Univ. Hawaii)

The Partnership for Extreme Xtallography (PX^2) is one of the newest COMPRES-supported experimental facilities for high-pressure Earth science research, having started during COMPRES-III. PX^2 is located at the Advanced Photon Source experimental station 13-BM-C at the Argonne National Laboratory, and is operated in collaboration between GSECARS, and the University of Hawaii. PX^2 offers excellent capabilities for conducting powder diffraction in diamond anvil cell, and offers novel and unique capabilities for more advanced single crystal experiments.

PX^2 focuses on X-ray diffraction-based high-pressure research using diamond anvil cells. Because of the unique instrumentation available in the new station (a 6-circle Newport diffractometer) experimental characteristics such as resolution/peak width and angular coverage are dramatically improved. Besides the focus on powder diffraction and total scattering experiments, PX^2 offers single-crystal experiments in diamond anvil cell, including laser heating of single crystals (currently still in commissioning).

The PX^2 facility opened doors for COMPRES users in February 2015. By the end of July 2016, 74 groups of users have carried out research at the PX^2, with 73% of them coming from COMPRES member institutions. The PX^2 instrument has been in high demand among the COMPRES users, as evidenced by high oversubscription rates. Usage of the 13BMC experimental station is currently split between surface scattering and high pressure research in approximate 2:3 proportion. During the 2016-3 APS run high-pressure program will have available 43 out of 65 total beam time days. All of the instrument time, except for time reserved for commissioning and testing of new components, is distributed based on APS General User Proposals (GUP), which are peer-reviewed by the APS Proposal Review Panel.

PX^2 offers multiple X-ray related techniques, including single crystal and powder diffraction, multi-grain diffraction, pair-distribution function measurement and thermal diffuse scattering. Several auxiliary techniques, including a resistive heating setup, membrane pressure controller, ruby fluorescence pressure determination and controlled chemical environments (humidity and oxygen partial pressure), are provided to PX^2 users. Users study a wide variety of samples, including minerals of the Earth's mantle and core, meteorite thin slices, quasi-crystals, organic materials and energy-related materials.

While the instrument became quickly operational, productive, and in high-demand during its first 2.5 years, we are still actively developing and commissioning new techniques. In the next COMPRES funding cycle, we will focus on the following technique developments.

High pressure in situ single-crystal X-ray diffraction with simultaneous laser heating

High-pressure powder diffraction combined with laser heating is one of the main and most successful experimental methods in mineral physics for measuring crystal structure, stability, and elastic properties at deep Earth conditions. However, the laser heating techniques have intrinsic limitations. At high temperature, the sample may recrystallize and grow, thus reducing the number of distinct grains, affecting the interpretation of powder diffraction patterns. Temperature gradients can cause mechanical mixing and chemical transport within the heated sample. Inhomogeneities of the temperature field often cause coexistence of high and low temperature phases and make it difficult to properly characterize crystal structures and phase boundaries. One approach to overcome these limitations is to take advantage of the high-intensity beam from insertion-device end-stations to make rapid measurements, minimizing complications that are related to transport processes in the laser-heated spots.

The PX^2 beamline offers an alternative approach by combining laser heating with single-crystal (as opposed to powder) diffraction techniques, which are well suited to a bending magnet station.

The new approach takes advantage a technique developed at the European Synchrotron Source (ESRF) in which the laser-heating optics are attached to the sample rotation stage. We are in the process of implementing rotating optics for laser heating of single crystals into the PX^2 beamline design.

Single crystal diffraction in natural inclusion-host systems
Some minerals derived from high pressure environments, such as diamonds, contain inclusions that provide information about the histories of their origins. The host crystals are usually chemically inert and physically strong, thus providing a nearly perfect container to preserve mineral inclusions from alteration or re-equilibration. Significant efforts have been devoted to characterization of the major and trace element chemistry of the inclusion minerals. One of the important lessons learned from previous inclusion-host studies is that the relationships between the histories and properties of inclusions, host crystals and the rocks, in which the hosts are embedded, are complex. The inclusion and the host crystals can crystallize either at the same time, or one after another. Understanding the formation scenario is critical for drawing conclusions about the paragenetic environment and conditions, particularly in the case of multiple inclusions in the same host. While we have a reasonable understanding of the mineralogy and major element chemical composition of the mantle, the oxygen fugacity is not nearly as well understood, particularly where redox reactions involving carbon are concerned. Natural inclusions in diamond provide good environment to examine the redox conditions of the mantle when the inclusions were crystalized. Good characterizations of the inclusions are needed in order to study the redox conditions at the time of crystallization. Previous analytical probes used to study inclusions that address the details of chemical composition and trace elements were usually *ex situ* methods. These require the inclusion to be either exposed or completely liberated from the host. The advantage of these methods is that more accurate information can be obtained via direct access to the sample; however, this is at the expense of the inclusion losing its original residual stress environment and jeopardizes the chemically insulating encapsulation. *In situ* methods, which probe the inclusion through the diamond, have to deal with more complex corrections and calibrations, but in return offer a fully non-destructive approach that can still provide very valuable information. Synchrotron micro-focus X-ray diffraction, which can be applied *in situ*, offer very good alternatives to exclusively *ex situ* experiments such as electron microprobe analysis (EPMA), but require some further developmental efforts to reach the required reliability and accuracy levels. We will incorporate the existing single crystal diffraction capability at PX^2 with the X-ray Absorption Near Edge Spectroscopy (XANES) and micro-tomography capabilities at GSECARS to study our samples. We plan to apply this methodology to study a suite of solid inclusions in preselected natural samples and derive reliable information about the composition, mineralogy, oxygen fugacity and water content of the mantle rocks.

4.1.3. APS Gas Loading for DAC (PI: Mark Rivers, Univ. Chicago)

The COMPES/GSECARS gas loading system at the APS has been a major advance for measurements of structure, phase stability, and equations of state at high pressures in the diamond anvil cell, which require a hydrostatic environment to accurately interpret the pressure dependence of these properties. Prior to COMPRES's installation and support of this system in 2008, the use of noble-gas pressure media was restricted to a small number of scientists with access to systems at the Carnegie Institution or at the Lawrence Livermore National Laboratory. The COMPRES/GSECARS system has completely transformed the quality of measurements requiring hydrostatic conditions, and Ne or He loading is now the norm for most synchrotron-based diamond anvil cell experiments around the world. The system is available to the entire community.

The system began operation in February 2008 and has been running almost continually with minimal downtime since then. The system works extremely well, with the only significant problems being some failures of the commercial compressor. In-house technical support is available to repair such problems, and the mean time to repair has typically been 1 day. We have recently purchased a spare compressor, so that we can rapidly swap it if there is a major problem.

One problem that has arisen recently has been a dramatic increase in the cost of neon in the US. The price has increased from about $3,000 for a 6,000 liter cylinder to over $22,000. The gas loading system typically has used 3 cylinders per year. We have solved this problem in two ways. First, we found a supplier of neon in the Ukraine who sells 7,500 liter cylinders for $3,050, or 9 times less than the US price per liter. We have recently purchased two cylinders from Ukraine. Even with an additional $2,000 in shipping costs the cost was about 5.5 times less than the US price. Second, a COMPRES-funded low-pressure compressor was installed in May 2016 which greatly reduced our neon consumption by allowing for more efficient extraction of Ne from the large gas cylinder, thus reducing waste.

The COMPRES/GSECARS 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 support from COMPRES allows the system to be available for users who cannot afford the time or money to travel to APS, by providing a “mail-in” service. It also allows the system to be available to users who are conducting experiments at APS sectors other than GSECARS. These include users from sectors 3 (inelastic), 4 (magnetism), 16 (HP-CAT), and 32 (imaging), 34 (microdiffraction), and others.

Personnel

This COMPRES proposal seeks funding for 50% of a staff scientist for the gas loading system. The responsibilities of the COMPRES supported portion of this position are 1) loading cells from the “mail-in” program, i.e. that are sent to the APS by users who do not travel here 2) training and assisting on-site users working at beamlines other than GSECARS with loading their cells. Dr. Sergey Tkachev began in this position in 2010. The other 50% of Sergey’s salary and responsibilities are covered by GSECARS. Guy Macha is a GSECARS funded technician who provides mechanical support for the system. We do not seek any support for Guy in this proposal.

Operations, Performance Metrics

In addition to this mail-in service, there were 640 diamond anvil cells loaded in this year. 199 of these cells were prepared for experiments at GSECARS, while the remaining 441 were prepared for experiments at other APS sectors, home laboratories and other synchrotrons. The gas loading of all 640 cells were directly assisted by Sergey. Thus on average he is assisting in gas loading of more than 2 cells every working day. The number of cells loaded per year has been approximately constant for the past 3 years, after growing rapidly in the years 2009-2012.

Sergey spends more than 60% of his time assisting such users and providing the mail-in service. Because of the constantly growing usage of the GSECARS/COMPRES gas loading system during the normal APS user operation periods, significant time is required for repairs and preventative maintenance for the valves and commercially built compressor. Sergey and Guy perform these tasks, during the downtime between the APS beamtime cycles.

Publications

Users of the gas loading system have reported 137 publications during the period 2011-2016. There have certainly been more than this, because not all users have reported in their publication that they used the gas loading system. At least 50% of the ~700 diamond cell publications from GSECARS and HP-CAT in this period have used the gas loading system.

Community/Broader Impacts

The success of the COMPRES/GSECARS system design has led other groups to copy many aspects of it for systems of their own, including HPSync at the APS. In addition four other identical units have been built by the University of Chicago Engineering Center. These have been sold to Jennifer Jackson at Caltech, the Advanced Light Source at the Lawrence Berkeley National Laboratory (where it is available to users of the COMPRES supported high-pressure beamline, 12.2.2), Sandia National Laboratory, and the LNLS synchrotron in Brazil. Sergey has provided valuable expertise and hands-on support during construction for these new gas loading systems. There are expressions of interest for additional systems, both in the US and abroad.

4.1.4. APS Nuclear Resonant and Inelastic X-Ray Scattering at Sector 3-ID DAC, Mössbauer (PI: Jay Bass, UIUC)

High-resolution inelastic X-ray scattering techniques provide the COMPRES community with access to a suite of synchrotron-based measurements of a variety of materials—especially iron-rich metals and oxides—at high pressures and temperatures. Because Fe is the primary constituent of the core, and a major chemical constituent of most mantle minerals, these techniques provide the Earth and planetary science community with unique opportunities to study the electronic, elastic, and thermodynamic properties as well as phase stability of iron-bearing core and mantle phases under extreme pressure and temperature conditions. Using experimental methodologies largely pioneered at Sector 3 of the APS, these techniques have recently been used to explore electronic high-spin low-spin transitions in lower mantle minerals (Jackson *et al*., 2005; Lin *et al*., 2008; Chen *et al*., 2012; Catalli *et al*., 2010, 2011), the melting points of Fe and Fe alloys at high pressure and the temperature of the core (Jackson *et al*., 2013; Zhang *et a*l., 2016), sound velocities in Fe and Fe alloys at high P-T conditions and the composition of the core (Lin *et al*., 2005; Chen *et al*., 2014), isotopic fractionation factors for Fe and the redox state of the crust and mantle (Dauphas *et al*., 2014), the nature of the heterogeneous core-mantle boundary region (Wicks *et al*., 2010), and even the texture and rheologic properties of the inner core (Mao *et al*., 2012). These first-order geophysical problems will continue to be pursued in the future, while new users will undoubtedly pursue new applications of these versatile techniques.

High-resolution inelastic X-ray scattering (IXS) is a synchrotron technique that is used to measure bulk acoustic wave velocities, Vp and Vs, and elastic properties of single crystal and powdered samples. A great advantage of IXS is the ability to measure bulk sound velocities Vp and Vs and elastic properties on opaque metallic samples at high P-T conditions. Such velocity measurements are currently problematic or not possible using home-lab-based techniques such as light scattering or ultrasonics. A main thrust is to measure velocities on candidate core materials at high P-T conditions (Mao *et al.*, 2012; Liu *et al.*, 2014; Liu *et al.*, 2015). Comparisons of such results with seismic velocity models of the core are extremely promising for placing tight constraints on inner core composition, thermal state of the core, and identifying the light element(s) present in the core.

Several different techniques are provided under this facility umbrella, including Synchrotron Mössbauer Spectroscopy (SMS), Nuclear Resonant Inelastic X-Ray Scattering (NRIXS), and momentum-resolved inelastic X-ray scattering (IXS). The unique electron bunch time structure and high x-ray flux above 10 keV makes the APS the only practical source for inelastic x-ray scattering in the USA. COMPRES Users have access to **i)** on-line SMS during the 24-bunch and hybrid-filling modes, **ii)** on-line NRIXS during the 24-bunch mode, **iii)** on-line IXS studies with access to the high energy resolution HERIX-3 and HERIX-30 instruments, based on GUP or PUP proposals. **iv)** In addition, this COMPRES Facility offers offline conventional Mössbauer spectroscopy (CMS). It is well known that in Fe-bearing minerals, the valence and site occupancy of Fe affects virtually all properties. The CMS facility is thus heavily subscribed in characterizing samples for other experiments, and run products. This project will make off-line Mössbauer spectroscopy available by mail-in or drop-in service. The COMPRES-supported staff member runs the experiments, analyzes the results, and helps to write necessary sections of publications.

Proposed Work: The tight focusing of X-rays at Sector 3 allows experiments to be performed up to 300 GPa and up to 4000 Kelvin with laser heating (λ=1064 nm). Our proposed Facility project will enable the COMPRES community to exploit these new and unique capabilities. We request partial (2/3) support for a scientist (Dr. Wenli Bi) to work at Sectors 3, 30, and the offline Mössbauer lab. A 1/3 salary match will be provided by the APS. Dr. Bi will be: 1) developing new high pressure capabilities at all facilities; 2) working with the COMPRES community in developing competitive proposals for beam time; 3) assisting COMPRES during their beam time; 4) performing on-line analysis of their results (which is formidable even for experienced users); 5) education and outreach through organizing workshops; 6) assisting users of the conventional Mössbauer spectrometer; 7) writing instruction manuals for the complex software used in analyzing results; 8) maintaining the offline Raman system. Dr. Bi will be a primary contact for the COMPRES community. Scientifically we will enable measurements of charge and spin state of iron bearing minerals, velocity of sound, shear and compressional moduli, components of the elasticity tensor, Fe and Sn isotope fractionation, and phase identification under the high P-T conditions corresponding to planetary interiors. The experimental capabilities to be made available at Sector 3 of the APS are relevant for geophysics, petrology, and the geochemistry of rocks, minerals, and meteorites.

There are still many aspects of the various IXS techniques that have unharvested capabilities. One example is potential applications to paleomagnetic measurements. The tight focusing and polarization of synchrotron X-rays should make it possible to identify the direction of magnetization in individual mineral grains as small as 10 µm in a rock, and to identify evidence for non-dipole components of Earth's magnetic field. Accurate polar reversal paths could be reconstructed back into the early Earth. Another area of development will be the application of the Mössbauer microscope with a few micrometer spatial resolution to meteorite research. Finally, high spatial resolution studies will play a role in characterizing new phases synthesized in multi-anvil and diamond anvil cells.

Proposed research topics:

1) High-Spin Low-Spin Transitions in Glasses and Melts: at the extreme P-T conditions of the deep mantle.
2) Velocity-Density Relations of Fe-Alloys leading to to new compressional wave velocity-density ($V_P-\rho$) models of Fe alloys as a means of determining the chemical composition of the core, including the identity of light elements.
3) Measurements of force constants in iron rich samples, which allow calculations of equilibrium isotopic fractionation factors between mineral phases. This is useful for interpreting Fe isotopic variations in low-temperature aqueous systems on Earth and possibly on Mars in the context of future sample return missions.
4) Similar measurements equilibrium isotope fractionation factors among Sn-bearing metals and oxides provide additional constraints on how terrestrial planets differentiated into metallic cores and silicate mantles.
5) Iron melting at high pressures: SMS studies in conjunction with laser heating in the diamond anvil cell at Sector 3 address fundamental questions such as the temperature at the center of the Earth. New methods were developed to identify the onset of melting while making more accurate temperature measurements, thus providing ways to test hypothesis about the temperatures throughout the Earth's core.

Proposed technical work for 2017-2022 period:

1) During this period, we plan to implement a Fast Chopper for SMS studies to boost the data acquisition rate by two orders of magnitude. This will open additional beam time and unprecedented possibilities for the Earth science community. The fast chopper will be the first of its kind.

2) A new x-ray focusing system for 3-ID-D station to focus down to a few micrometers. This will enable very high pressure measurements and an imaging microscope.

3) Implementation of new array APD detectors to improve the time resolution and count rates.

4) Developing polarization based crystal optics for rapid determination of magnetic phase transition boundary as a function pressure.

Equipment for the above-mentioned projects are funded by the APS Operational funds. The COMPRES-funded scientist will play a crucial role in implementing this technical agenda.

4.1.5. Multi-Anvil Development/Cell Assembly Program (PI: Kurt Leinenweber, ASU)

The COMPRES Multi-Anvil Development/Cell Assembly Program (MADCAP) is well-established at ASU and has many successful collaborations with COMPRES-affiliated multi-anvil laboratories in the United States and around the world. The self-supporting part of the program has developed a series of cell assemblies that are in wide use for multi-anvil and DIA devices, both in home laboratories and at beam lines. It provides a framework for laboratories to share information about materials, techniques, and the pressure/temperature calibrations of the assemblies, and makes it possible for newly built laboratories to more easily implement the latest in multi-anvil technology.

For the next five years of COMPRES, we have a number of important targets for improvement of existing and development of new multi-anvil capabilities.

Improvements to the Standard 6-8 Assemblies: The standard 6-8 assemblies are the multi-anvil cell assemblies based on the octahedral pressure medium design (see above Figure) for which assemblies have been standardized and are widely and reliably used. The 18/12 assembly will be improved to provide a more uniform temperature gradient within the sample area – this will be added to the assembly list as a new assembly alongside the existing one which will still be available. The development of the largest size, 25/15 assembly, will then be completed and the assembly made available for large-volume synthesis at and below 6 GPa. A mullite pressure medium will allow the 25/15 to be thermally efficient and inexpensive compared to using a zirconia sleeve (which is unnecessary below 6 GPa pressure where mullite is stable and a good thermal insulator).

Higher pressure: The MADCAP program will become involved with the new DELVE initiative led by Yanbin Wang and Tony Yu at GSECARS, described elsewhere in this document. This initiative will extend the pressure range of multi-anvil assemblies to 50 GPa from our current typical operating limit of 25 GPa. We will assist the DELVE initiative in developing the smaller cell assemblies and in procuring and testing carbide for the project, which requires harder grades of carbide than currently commonly in use. The new, smaller assemblies will be made available through our project as standardized cell assemblies once the development is complete. MADCAP collaboration will be crucial for the success of this program and we look forward to contributing to it.

DIA assembly program: We have been assisting in the development of DIA (cubic-style pressure medium) assemblies with several collaborators involved in beam line operations, including Matthew Whitaker, Paul Raterron, Pamela Burnley and others. Several standard DIA assemblies are now available from this effort. In the next five years, we will work on improvements to these assemblies and the creation of better standard DIA assemblies will be the ultimate target. Because of size limitations and the need for extra parts for deformation and ultrasonic measurements, many of the DIA assemblies developed so far have steep thermal gradients and difficulty in locating thermocouples close enough to the sample. Although this is a challenging area, we will work on design improvements to the DIA assemblies to provide a sample with reduced thermal gradients and better thermocouple placement and performance, while still allowing deformation pistons or ultrasonic buffer rods to reach the sample.

Larger volumes: The MADCAP program has committed to assisting in the development of larger-volume assemblies for reaching high pressures with significantly larger samples (for example, a centimeter-sized sample at 18 GPa). This effort will follow the acquisition of a 5000-ton press in the United States. The program will assist any COMPRES laboratory that is developing such a press, regardless of where it would be located.

Community inclusion: The COMPRES cell assembly development program is well-known in the community and has been able to create good levels of trust and communication between otherwise competing laboratories on the particular issue of assemblies and techniques. We would like to take more advantage of this good will by promoting more communication between the laboratories and establishing more documentation on interlaboratory calibrations, assembly materials, design specifications, and techniques for successful multi-anvil experiments. The product will be several openly available documents including pressure calibration charts, temperature calibrations, materials and sources, and instructions. Some of this is already available but the new documentation will include input from many users, and will cover a wider range of the existing assemblies and even have descriptions of some of the ones that are in development. The communication between laboratories will be increased using the project as a hub for interlaboratory interaction.

Conclusion: The MADCAP has produced over time numerous advances in multianvil technology and in addition has sustained a widely-used program of distribution of calibrated and dependable cell assemblies that has benefitted numerous labs worldwide. We will continue this program in ways that are crucial for the further development of multianvil technology.

4.1.6. NSLS-II Beamline Frontier Infrared Spectroscopy (FIS) Facility for Studies under Extreme Conditions (PIs: Russell Hemley, Zhenxian Liu, GWU)

Understanding the behavior of Earth and planetary materials at extreme *P-T* environments is crucial for developing comprehensive insights of the structure, dynamics, and evolution of planetary interiors. High-pressure spectroscopy from far-infrared (IR) to ultraviolet (UV) provides essential and often unique information about the properties of such materials under these conditions. In particular, vibrational IR spectroscopy provides detailed information on bonding properties of crystals, glasses, and melts, thereby yielding a microscopic description of structure, phase transitions, and thermochemical and electronic properties. Infrared synchrotron radiation is an ideal source for coupling with diamond anvil cell (DAC) techniques and provides a tremendous improvement in flux with well-collimated far- to near-IR beams and giving diffraction-limited spatial resolution with unmatched signal-to-noise.

The high-pressure infrared program at the National Synchrotron Light Source (NSLS) was initiated by our group in 1990. Since then the program has become an important resource for the high-pressure user community, emerging from a single group program to the first dedicated high-pressure synchrotron IR facility (NSLS-U2A) in the world, with the capacity for synchrotron IR micro-spectroscopic techniques, Raman scattering, and visible spectroscopy combined with diamond-anvil cell methods for measurements of far- to near-IR and visible spectra of materials from ambient to ultrahigh pressures at variable temperatures. With funding from COMPRES since 2002, NSLS-U2A has become a unique and important resource for the consortium and a COMPRES showcase for the worldwide earth science and high-pressure research communities.

The legacy of success at NSLS-U2A led to the development of the Frontier Synchrotron Infrared Spectroscopy Beamline under Extreme Conditions (FIS) at NSLS-II, the successor of U2A, with funding from the NSLS-II FY16 operation funds for procuring the storage ring optical extraction system and construction of laboratory space (beamline cabin) on the NSLS-II experimental floor. This beamline will open new opportunities for our high-pressure user community by taking the advantage of the state-of-the-art IR facilities at NSLS-II. Funding support through COMPRES IV will enable us to extend high-pressure IR programs and will be crucial for continued access for the COMPRES user community to the world-class synchrotron IR facilities at NSLS-II. As such, the facility will be able to address major new problems in high-pressure Earth and planetary science – from outer solar system bodies to the Earth's core.

The Frontier Infrared Spectroscopy (FIS) Beamline at NSLS-II

NSLS-II is the newest state-of-the-art 3 GeV electron storage ring in the United States. Based on a storage ring design current of 500 mA, top-up injection and the overall ultrahigh stability of the facility, performance estimates indicate that NSLS-II will be world-leading in terms of brightness and signal-to-noise over the widest possible spectral range. The spectral range includes the very far-infrared which is crucial for uncovering novel behavior in systems such as dense hydrogen and other planetary gases, hydrogen bond symmetrization in hydrous minerals, insulator-metal transitions, superconductivity, ferroelectricity and magnetism, and investigations of the full range of Earth and planetary materials as well as materials ranging from semiconductors, metals, energetic materials, high-explosives, plastics, and complex composites.

As one of the five beamlines developed as part of the NSLS-II BDN beamline suite, FIS initially will share the first synchrotron IR beam with the Magnetic, Ellipsometric & Time-resolved Infrared Spectroscopy (MET) beamline. The NSLS-II has committed \$2M from its FY16 operation funds for two major items of the beamline construction, of which \$1.1M was devoted to the storage ring optical extraction system and \$0.9M to the end-station laboratory space (beamline cabins) on the experimental floor. The procurement of the optical extraction system has been completed and the synchrotron IR beam is scheduled to be delivered to the end stations in May, 2018. The design and construction of the beamline cabins, which have more than four times the floor space of NSLS-U2A, are underway and beneficial occupancy is expected in spring, 2017. With its smaller source size, higher brilliance, and broader spectral range, NSLS-II ideally matches the materials research under extreme *P-T* conditions through the coupling of the diamond anvil cell techniques. The execution and realization of FIS will provide superior ability to continuously accommodate a growing user community and further expand geoscience research at the NSLS-II.

New Techniques and Opportunities at FIS for the High-Pressure Geoscience Community

A major goal of Earth and planetary science is the extension of experimental techniques to still higher temperatures and pressures in order to directly probe thermodynamics, chemical diffusion, and molecular coordination *in-situ* under conditions of deep planetary interiors. This requires brighter sources with diffraction-limited performance and superior stability of the IR source. The large-gap dipole at the NSLS-II will provide an ideal IR source high *P-T* DAC experimentation. Given the excellent pace of current construction of FIS, our priority for year one is to relocate all existing and new end-station equipment into the beamline cabins, develop advanced user friendly/routine external and laser heating DAC techniques, and to begin *in situ* high *P-T* measurements on variety of Earth and planetary materials. This plan will allow us to accommodate science commissioning on day one when the IR beam is extracted and delivered to the beamline cabin. With these new techniques implemented at NSLS-II, FIS will provide invaluable set of tools for the COMPRES user community to address numerous scientific questions in Earth and planetary science and beyond. Major research areas include:

a) *Earth and Planetary Sciences*: mimic the mantle extreme conditions and study the Earth's deep water cycle: IR spectroscopy is a unique probe due to its exceptional sensitivity to the signature of volatile components. As such, it serves as an invaluable tool for evaluating the behavior of hydrous and nominal anhydrous minerals at high *P-T* conditions;
b) *Deep Carbon Cycle Research*: study behavior of carbon-bearing materials in Earth's deep interior conditions by vibrational spectroscopy;
c) *Material Sciences*: study the metallization of hydrogen and hydrogen-rich materials such as H_2S and methane under extreme high pressure and low temperature;
d) *Energy Science*: Optical studies of nano-crystalline materials and porous materials including BN nanotubes and zeolites/natrolites under extreme conditions for energy applications;
e) *Dynamic Compression*: probe material behavior on short time scales combined with the pulsed synchrotron radiation.

Overall, the joint effort by NSLS-II and COMPRES for FIS is very cost efficient. The total cost for the first synchrotron IR beam extraction and beamline cabins is over $5M, fully covered by NSLS-II. The requested funds will allow us to not only maintain the routine beamline operation but also continue to develop the advanced techniques such as time-resolved IR spectroscopic methods for studies under dynamic compression or fast cooling to tackle these key questions and challenges for the user community throughout the COMPRES IV period and beyond.

4.1.7. NSLS-II XPD & APS 6BM-B Beamlines MAP (PI: Don Weidner, Stony Brook)

The COMPRES multi-anvil beamline program has been built around the need to understand mechanical properties of the materials within the Earth at the relevant pressures and temperatures The research focuses on rheology--measurements of elastic, anelastic, and plastic properties of mantle minerals at extremes in pressure and temperature. In addition to our pioneering rheology capabilities, the user facility can also perform a variety of other studies, including measurements of equations of state and phase stability at simultaneous high pressures and temperatures.

The coupling of synchrotron-based X-ray diffraction with large-volume presses has revolutionized studies of the rheological properties of materials at high pressures and temperatures. Deformation measurements rely on radiography showing how sample length changes as a function of time, for a given applied stress. The high energy, high intensity synchrotron X-ray source permits both imaging via radiography to determine macroscopic changes in the sample size, and therefore macroscopic strain, while also measuring mineral structure and lattice parameters, providing information about lattice elastic strain. Acoustic wave speed measurements by *in situ* ultrasonic techniques benefit from X-ray radiographic determination of sample length. Studies of frequency dependent elastic properties and attenuation require time-dependent measurements of sample stress and strain amplitudes in a sinusoidal stress field.

The technology was first developed in Japan with the DIA (a cubic multi-anvil high-pressure device that generates differential force, unlike the Kawaii-type press based on truncated cubes that distribute a force uniformly on the faces of a sample octahedra). The DIA geometry provided an opening so that it could be used in conjunction with a synchrotron source that enabled x-ray analysis of the sample [*Shimomura, et al.*, 1985]. The DIA was followed by the development of the deformation DIA, (the D-DIA), which was conceived by Weidner and developed by Weidner, Wang, Getting, and Durham [*Durham, et al.*, 2002; *Wang, et al.*, 2003]. The D-DIA system was first used on the COMPRES beamline at the NSLS and has been copied around the world at most major synchrotrons. Subsequent developments within and outside COMPRES have led to new capabilities in studying high pressure mechanical properties:

- Analysis of stress using x-ray diffraction [*Singh*, 1993];[*Mao, et al.*, 1998].
- Analysis of strain from x-ray images [*Vaughan, et al.*, 2000]; [*Li, et al.*, 2003]. X-ray shadow images such as illustrated in the Figure shows a sample sandwiched between two metal foil discs. The radiography technique allows direct measurements of the sample length, thus opening up the possibility of measuring acoustic velocities.
- Measurement of ultrasonic acoustic wave travel times in a sample (developed by Liebermann and Li and used for the first time at the COMPRES beamline at the NSLS). The added information of sample length led to the ability to measure sound velocity at elevated pressure and temperature.
- Use of x-ray transparent anvils in the multi-anvil system in order to obtain the necessary diffraction data for stress analysis [*Chen, et al.*, 2004.; *Li, et al.*, 2004].
- The understanding of the effect of plasticity on X-ray stress measurements. [*Li, et al.*, 2004 ; *Weidner, et al.*, 2004].
- The conception, design, and construction of the rotational Drickamer anvil system (RDA) (Karato) and its implementation at the COMPRES beamline at the NSLS. The RDA system rotates the sample to develop strain, is capable of reaching lower mantle pressure, and very high strains.
- Implementation of conical slits to allow white energy-dispersive [*Chen, et al.*, 2004; *Li, et al.*, 2004; *Li, et al.*, 2004]. The slit system limits the volume of sample that scatters x-rays to the detector, yet provides sufficient coverage of the sample for determination of the orientation and magnitude of the stress field. Thus, the measured spectra can be free of complex sample container and the high pressure medium.. Use of the RDA system requires white x-rays and the conical slit system to isolate the sample region with a specific stress orientation.
- Weidner designed a deformation multi-anvil system based on the Kawai geometry to reach higher pressure than the DIA system for a given sample volume. The DT system was first applied to a miniature version, the DT-10 that used 10 mm cubes. It has been generalized to the DT-25 that uses 25 mm cubes – the standard size currently used in systems such as the Walker module. The guideblocks for DT-25 are being installed at the new Brookhaven synchrotron, the NSLS II.

In October, 2014 the NSLS was shut down and the NSLS II began operations. The new synchrotron offers capabilities that enable improvements in imaging, spatial resolution, and energy resolution. However, it will be at least a decade before the full capability of the new ring is realized. To maintain the experimental capability, Weidner et al. obtained space and permission to install and operate a multi-anvil high pressure system at one of the first-generation beamlines, XPD (x-ray powder diffraction). It will operate in monochromatic mode at moderately high energy (optimized at 50 keV). The XPD will house a DT-25, a Kawai style compression system with an additional deformation mode, in conjunction with at 1000 ton press. It will be the first such system installed at a synchrotron and is designed for deformation experiments employing a uniaxially-applied differential stress at hydrostatic pressures up to 30 GPa, allowing access to lower mantle phases. A standard D-DIA apparatus can also be used at XPD. Deformation experiments and ultrasonic velocity measurements will both be possible at the XPD beamline.

In October, 2015 a DDIA facility at beamline 6BM of APS was opened to users. It provides white x-rays with about 1/5 of the NSLS flux. This system is designed for deformation experiments (up to about 10 GPa) and ultrasonic acoustic velocity studies.

4.2 New Facilities proposed for COMPRES IV

4.2.1. Community Extreme Tonnage User Service (CETUS): A 5000 Ton Press Open Research Facility in the United States (PI: Lisa Danielson, Jacobs Technology, NASA Johnson Space Center)

Multi-anvil apparatus designs, rated to 5000 tons or more, allow for relatively large experimental sample volumes at a given pressure and have contributed to the exploration of deep planetary interiors, synthesis of ultra-hard and other novel materials (Frost et al., 2004; Liebermann, 2011; Isobe et al., 2013). However, no such facility exists in the United States. We propose to establish the Community Extreme Tonnage User Service (CETUS), as an open user facility for the entire high pressure research community. CETUS will feature a 5000 ton multi-anvil press, supported by a host of extant co-located experimental and analytical laboratories and research staff.

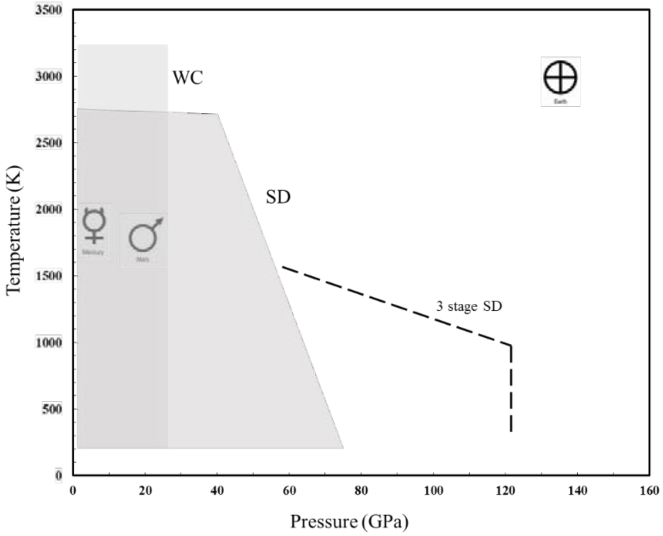


Figure 4.2.1.1. Schematic of achievable pressure-temperature space of multi anvil experiments with various second-stage anvils, modified after Lieberman (2011). Shaded block labeled “WC” (tungsten carbide) is the current pressure temperature regime of multi-anvil apparatuses in the United States. “SD” is sintered diamonds, which require modifications to the pressure module normally used for WC anvils. Even higher pressures have been achieved by use of a 3 stage assembly with additional nano-polycrystalline diamonds, Kunimoto et al. (2008). Symbols for Mercury, Earth, and Mars approximate the core-mantle boundary conditions. See Voggenreiter technical

A high-tonnage press has a number of potential benefits to expanding high pressure research. Larger sample volumes will allow better control of the sample environment and complex mixtures of starting materials to be studied in greater detail, contributing to a greater understanding of physical properties of planetary interiors (e.g., thermal conductivity), rheology, paleomagnetism, all of which are linked by complex early planetary dynamics. This new capability even opens experimental opportunities for studies of the evolution and mantle-core compositions of exoplanets such as super-Earths (Fig. 4.2.1.1).

A large sample volume at lower mantle conditions will enable research on planetary scale processes relevant to life on Earth, such as minor phases in the mantle, particularly volatile-rich phases, allowing a better understanding of water and carbon cycling. It will serve an identified need for a user-facility for high-pressure sample synthesis. Synthesized samples will be curated and shared among researchers for multiple applications, such as shock wave

Figure 4.2.1.2 Team members of Experiments in Extreme Environments Laboratories (EEELs), who will augment support for CETUS, including fabrication and development. From center-front clockwise: Lisa Danielson[1], PI, HPXL lab manager; Kellye Pando[1]; Loan Le[1]; Roland Montes[1]; Jenny Rapp[1]; Mark Cintala[2]; Dave Draper[2]; Frank Cardenas[1]; Francis McCubbin[2]; Poorna Srinivasan[3]; Kathleen Vander Kaaden[3]; Mya Habermann[1,3]; Kevin Righter[2]; Ian Szumila[1,3]. [1]JETS Contract; [2]NASA civil servant; [3]graduate student. Not shown: John Jones[2], Fred Hörz[1], emeritus; Etienne Medard, visiting scientist, LPI; Asmaa Boujibar, NPP postdoc.

experiments, trace element and isotopic chemical analysis, and further characterization with neutron and/or X-ray diffraction. Finally, materials synthesis at high pressure for practical applications requires equipment that produces these materials in a scalable way. For example, it would be desirable to synthesize one-dimensional nanostructures of ultra-hard materials by hard-templating techniques, integrate these structures into other materials for their mechanical reinforcement, and then test the mechanical properties of these materials.

The Experiments in Extreme Environments Laboratories (EEELs) are available to support the construction and operation of the proposed CETUS facility (Fig. 4.2.1.2). EEELs offer a wide range of complementary and/or preparatory experimental options. Any required synthesis of materials or follow up experiments can be carried out using controlled atmosphere furnaces, piston cylinders, multi-anvil, or experimental impact apparatus. Additionally, the NASA JSC division houses two machine shops that would facilitate any modification or custom work necessary for development of CETUS, one for general fabrication and one located specifically within our experimental facilities. Also available is a general sample preparation laboratory, specifically for experimental samples, that allows users to quickly and easily prepare samples for e-beam analyses and more.

A service to be offered to COMPRES community members in general, and CETUS visiting users specifically, is a multitude of analytical instrumentation literally steps away from the experimental laboratories (see table below). This year site funding pursued to support the EEELS laboratories through NASA's Planetary Science Directorate, which should result in substantial cost savings to all visiting users, and supports EEELS's mission of interagency cooperation for the enhancement of science for all.

Ebeam Suite Instruments	**Other Microanalyses**	**Additional Laboratories**
FEI-Quanta FIB	ICP-MS	XRD
JEOL 8530F, Cameca SX-100 EPMA	TIMS, GC+Quad MS	Mössbauer
JEOL 7600 FE, 5910 LV SEM	Raman	Nanoscale 3D printer
JEOL 2500 SE, 2000 FX TEM	FTIR	TG, DCS
NanoSIMS	L^2MS (organics)	Soluble organics

The proposed CETUS facility will be a major undertaking, requiring significant managerial and technical dedication to its success. To ensure long term financial health of CETUS, we will pursue a diversified funding portfolio. The PI is in a unique position as an employee of Jacobs Technology to draw funding from multiple sources, including those from industry and commerce. On June 3rd, 2016 a Planetary Major Equipment proposal was submitted to the NASA Emerging Worlds solicitation, requesting the full cost of the 5000 ton press and additional FTE for the project management. The PI anticipates that industry will play a greater role in funding ongoing operations through research and development partnerships, and the PI will be requesting such through Jacobs in the coming weeks. Following the model of collaborative success that COMPRES has historically brought to the experimental community, support will be requested for COMPRES and NSF to fund two full time researchers, who will be committed full time in effort to the development and operation of CETUS for the lifetime of the facility.

4.2.2. Community Large Multi-Anvil Press Facility (LMAPF) at ASU through COMPRES (PI: Kurt Leinenweber, ASU)

The PI proposes to build a national multi-anvil facility by combining a new 6000-ton press together with existing two 1000-ton presses, two 300-ton presses, and other high-pressure equipment at Arizona State University. The large multi-anvil press facility (LMAPF) will serve the COMPRES community by supporting users in conducting experiments and synthesizing high-quality samples at deep mantle pressure-temperature conditions in a large multi-anvil press facility (LMAPF)

Figure 4.4.2.1 LMAP at Ehime, Japan

Plan for a community-serving facility

The proposal seeks to acquire a 6000-ton press to achieve higher pressure-temperature conditions for large volume samples than currently possible in the US. Instead of copying the existing capabilities in Ehime (Japan) and BGI (Bayreuth, Germany), the facility will be optimized for achieving the broadest possible benefits for the US high pressure community by taking full advantage of ASU's 20+-year experiences in managing multi-anvil user facilities and developing standardized and special purpose multi-anvil cell assemblies for the community. The project can help the open and diverse US community catch up and excel in the development of this critical technology, which has been led by the Japanese and European communities.

Examples for scientific and technical impacts

1. Preparation of large (up to ~1 cm^3) samples of transition zone and lower-mantle minerals for high-quality structure and property measurements, such as neutron diffraction, shock compression, deformation, elasticity, calorimetry, diffusion, rheology, and electrical conductivity, etc. Some of these measurements have been impossible due to the sample size limitation in the multi-anvil press.
2. Use of large sample assemblies to control oxygen fugacity and other thermodynamic properties of mantle transition zone and lower-mantle minerals and, importantly, complex multi-mineral phase equilibria.
3. In situ experimentation on large samples of mantle minerals – for example ultrasonic and elastic properties (seismic velocity), diffusion, electrical conductivity, and rheology, etc. The ability to perform such measurements is often limited by the sample volume. The LMAPF will facilitate major advances in *in-situ* measurements at high pressures and temperatures.
4. Synthesis of single crystals, which are uniquely suitable for measurements of any property that depends on direction in the crystal, including velocities, structural properties, and diffusivities.
5. Synthesis of novel ultrahard materials to enable higher pressure experimentation. For example, polycrystalline nanodiamond can be synthesized using the proposed LMASP and it has the potential to expand the pressure range of multi-anvil devices and diamond-anvil cells.

This capability will also lead to invention and synthesis of other novel materials and has potential for collaborations with materials scientist partners in industry and academia.

6. Preparation of relatively large volumes of materials to enable sharing of samples by multiple groups for interlaboratory comparisons and calibrations. This work can test and improve reliability and reproducibility of difficult measurements.

Why ASU?

Since the beginning of COMPRES in 2002, ASU has a long history of community-based development of materials and technologies for multi-anvil cell assemblies. Through the long-term efforts of Leinenweber with advice and assistance from Sharp and Tyburczy, and now others in the Advisory Committee, ASU is highly-regarded in its reputation for multi anvil capability combined with community service and communication. The 'COMPRES Multi Anvil Cell Development' project is the longest-running non-beamline project supported by COMPRES. Materials produced by ASU have been (and currently are being) used by scientists all over the world owing to their reliability, reproducibility, and traceable calibrations (see Leinenweber et al., 2012). Till and Shim are now heavy users of the expertise and capabilities of the ASU Multi Anvil facility, and the facility also hosts users from outside universities on a regular basis. The spirit of supporting the needs of the broader community sets ASU multi-anvil efforts ahead of other user facilities. This range of capabilities is also attractive to industrial users who need exploratory high-pressure work, additional expertise, or more accurate pressure-temperature determinations to assist with their industrial processes. The ASU multi-anvil lab already has ongoing research collaboration with Sandvik Hyperion in Ohio, for example.

ASU also offers a wide array of supporting facilities, including powder and single-crystal x-ray diffraction, Raman and IR spectroscopy, electron probe microanalysis, NMR, scanning electron microscopy and transmission electron microscopy. In addition to including the instruments that are currently in frequent use for high-pressure research there are also many other techniques available and users would be able to potentially break new ground with facilities that have not been highly utilized for high-pressure studies (such as SIMS and Nano-SIMS). In addition, there are a state-of-the-art diamond anvil cell lab (Shim) and end-loaded piston-cylinder lab (Till) that could be accessed through collaboration (these are PI-operated laboratories and not user facilities). Shim and Till are willing to play a vital role in developing the LMAPS and will serve as liaisons for the broader diamond-anvil cell and piston cylinder high pressure communities.

4.3 New EOID projects proposed for COMPRES IV

4.3.1. A Career Path for African-American Students from HBCUs to National Laboratories (PI: Bob Liebermann, Stony Brook)

The geosciences have the lowest diversity of all the science, technology, engineering and mathematics [STEM] disciplines. To address this poor record, the NSF Directorate for Geosciences established a new program entitled "Opportunities for Enhancing Diversity in the Geosciences" In 2011, we created a new initiative at Stony Brook University: "A Career Path for African-American Students from Historically Black Colleges and Universities [HBCUs] to National Laboratories."

The goals of this new initiative are to: Recruit undergraduate science and engineering students from underrepresented groups that received their undergraduate degrees from minority-serving institutions into the graduate program in the Department of Geosciences at Stony Brook University; Educate these student trainees through formal courses and research projects to the M. S. in Geosciences Instrumentation; Provide these student trainees with a marketable skill set in an emerging field between science and technology; Prepare these student trainees for employment as science associates in national user facilities of the U. S. Department of Energy [DOE], such as the National Synchrotron Light Source [NSLS] at the Brookhaven National Laboratory [BNL].

The M.S. in Geosciences Instrumentation program includes both formal courses in the Department of Geosciences and internship research conducted at the X-ray beamlines operated at the NSLS of BNL by COMPRES. The internship research projects typically involve the study of the physical and chemical properties of minerals under high-pressure conditions, which is the major theme of the COMPRES consortium. This new M. S. program addresses well-recognized low levels of participation by African-American students in the geosciences and is focused on fostering development and training of the diverse scientific and technical workforce required for 21st century geoscience careers. We are particularly interested in attracting women in view of the known gender inequality in science, technology, and mathematics, engineering [STEM].

Our program builds on the longstanding relationship between professors from Historical Black Colleges and Universities [HBCUs] and BNL. An outcome of this relationship has been the creation of an Interdisciplinary Consortium for Research and Educational Access in Science and Engineering [INCREASE], an organization whose main role is to support and advance research involving national laboratories and thereby to afford access to research facilities not available to HBCU faculty at their home institutions; our colleague Gabriel Gwanmesia from Delaware State University is a founding member of INCREASE. We have reached out to our colleagues at INCREASE institutions to help us identify and recruit students. In addition to INCREASE and the Photon Sciences Division of BNL, partners in this new initiative include: the Center for Inclusive Education and the Graduate School of Stony Brook University. All of these organizations have provided matching funds to compliment the NSF funding from Geosciences for the period 2011 to 2016.

To date, this program has graduated four M. S. students; Ashley Thompson and Adairé Heady from Delaware State University, Melissa Sims from the University of South Carolina, and Brandon Rhymer from the U.S. University of the Virgin Islands.

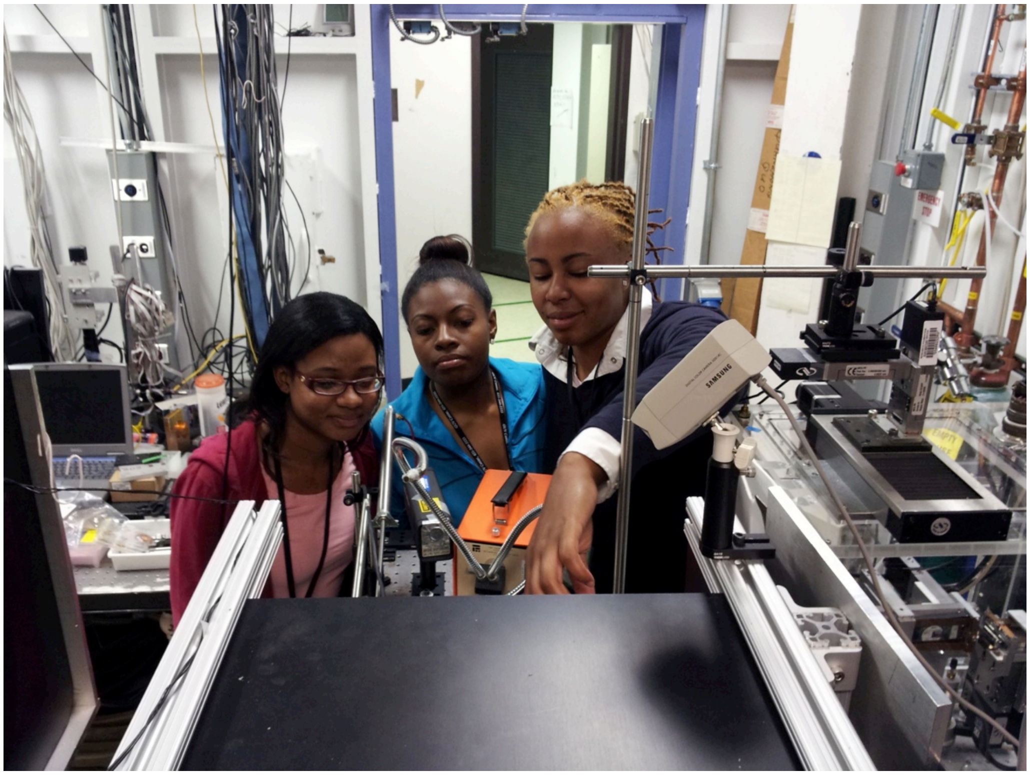


Fig. 4.3.1.1. Melissa Sims, Adairé Heady and Ashley Thompson at NSLS.

Melissa Sims, the student mentioned above who is now enrolled in the PhD program in Geosciences and has been awarded a prestigious W. Burghardt Turner Graduate Fellowship by the Center for Inclusive Education and the Graduate School at Stony Brook. Jesse John from Brooklyn College also holds a Turner Fellowship and is pursuing a PhD in Geosciences under the supervision of Professor John Parise. Funding from our diversity program will provide the matching and supplemental funding for both Jesse and Melissa as they proceed in their doctoral research. In May 2014, our program "A Career Path for African-American Students from HBCUs to National Laboratories" received the Dean's Award for Excellence and Innovation in Graduation Education at Stony Brook University. The NSF Directorate for Geosciences has approved a no-cost extension of our grant and both Brookhaven National Laboratory and the Graduate School at Stony Brook have extended their matching funding for another two years to August 2016. Unfortunately, the NSF Directorate for Geosciences has temporarily suspended funding for the this outreach program, so we are seeking new funding from COMPRES to extend the program beyond 2016. This proposal to the Education, Outreach and Infrastructure Development Program of COMPRES is for $180,088 for a two-year period from June 2017 to May 2019 to extend our diversity initiative for another two years. These funds will provide stipends and tuition support for two students to pursue a M. S. in Geosciences Instrumentation. We have requested matching funds from BNL [$50K per year for two years--pending] and the Graduate School [$10,870 per year for two years--approved]; these matching funds will enable us to educate an additional graduate student over this two-year period.

4.3.2. Infrastructural Development for Deep-Earth Large-Volume Experimentation "DELVE" --An EOID initiative for doubling pressure capability of large-volume apparatus

in the U.S. (Yanbin Wang, Tony Yu (GSECARS, APS), Donald Weidner, Matthew L. Whitaker, Baosheng Li (SBU, NSLS-II), Kurt Leinenweber (ASU), Zhicheng Jing (CWRU), Bin Chen (U Hawaii, Manoa), Yingwei Fei (CIW), Charles Lesher (UC Davis), Shun-ichiro Karato (Yale)

Recently, new tungsten carbide products have become available that provide the capabilities for achieving lower-mantle pressures using existing multi-anvil presses equipped with new types of sample assemblies. The Fujilloy TJS01 carbide is reported to reach 48 GPa using 1.5 mm TEL with tapered cubes (Kunimoto et al., 2016). This new nano-grained tungsten carbide has extremely high Vickers hardness of 2700 N mm^{-2} and possesses an excellent transverse rupture strength of 2.6 GPa (Wada, 2015). We have contacted the manufacturer (Fuji Dies Co., Ltd) and received quotations. The price of 14 mm TJS01 anvils is ~1/8 that of sintered diamond anvils. The low electrical resistance of tungsten carbide also makes it promising to conduct melting experiments at ~50 GPa range.

Here we request funding for a two-year project, in a concerted community effort to double the pressure capability of user-facility multianvil presses to ~50 GPa. The requested funds will be used to support users to GSECARS and NSLS-II beamlines to conduct DELVE experiments, with three specific measurement goals: lower mantle elasticity, viscosity, and petrology (descriptions of the science projects are given in the next section). We hope that by the end of this two-year support, we will obtain some preliminary results on the scientific projects, with several cell assemblies readily tested. These assemblies will then be made available to general users via ASU's MADCAP facility. The scientific results will be used to jump start an "ultra-high" pressure multi-anvil grand challenge proposal to the NSF.

Below we identify three major scientific goals to guide this collaborative development project:
Elasticity of the lower mantle. The main emphasis is to measure acoustic velocities of major minerals of the lower mantle, bridgmanite and $CaSiO_3$ perovskite. We plan to develop cell assemblies for ultrasonic techniques to investigate the elasticity of bridgmanite and $CaSiO_3$ perovskite to ~50 GPa and 2000 K.
Melts in the lower mantle. Structure and properties of silicate liquids with significant amounts of six-coordinated structural motifs are poorly known. These liquids may have played an important role in Earth's early history. Here we plan to develop cell assemblies for studying melting relations, density, and viscosity of silicate and Fe-rich melts up to 50 GPa.
Deep earth petrology. Here we are mainly concerned with interaction between lower mantle mineralogy and subducted lithosphere. The high aluminum content in slabs may affect phase relations in the surrounding mantle, thereby lifting or depressing the local 670 seismic discontinuity. Studies on the stability of dense hydrous magnesium silicates in the subducted lithosphere under lower mantle conditions would provide important information on the cycling of water in Earth.

Facilities Involved

Each of the project PIs has access to multianvil capabilities at their home institution. The majority of high-pressure modules in the offline labs use 25.4 mm cubes, similar to the T-25 at GSECARS. It is feasible to transfer pressure-load curves calibrated at beamlines to offline labs.

Online MAP systems are available at both APS and NSLS-II. The 1000 ton press at GSECARS beamline 13-ID-D has both white beam and monochromatic beam capabilities. Switching between mono and white beam has been made very easy with the recent modification on the monochromator design. Two multi-anvil modules can be used in the press. The T-25 module has been used extensively for the past ~17 years with energy-dispersive X-ray diffraction. This is identical to offline multi-anvil laboratories in the U.S. 25.4 mm Fujiloy TJS01 WC cubes are ideal starting point in this module, especially if the cubes need to be tapered to push toward 50 GPa (Kunimoto et al., 2016). Standard COMPRES beamline assemblies have been used (Leinenweber et al., 2012). These cells are the ideal starting point for this project. The DDIA-30 module has been vigorously tested both in deformation mode and in the double-stage mode. Both energy-dispersive and angle-dispersive x-ray diffraction can be conducted in this module. With 14 mm sintered diamond cubes as second stage anvils, pressures up to 35 GPa have been reached at 1800 K, with 1.5 mm TEL at a load of 350 ton (Wang et al., 2010). $LaCrO_3$ and TiB_2 were tested as heater materials; both worked satisfactorily. SD anvils were recovered intact after a series of tests to 350 tons, demonstrating the reliability of the system.

The new synchrotron at NSLS II is now operational. A 1000 ton press is being installed at the XPD beamline. It is expected to be open for commissioning by the end of the summer, 2016. This press will operate a DT-25 high pressure module, similar to the T-25 with the addition of independent, uniaxial compression along the axial direction of the press, providing the standard ‘hydrostatic’ pressure field or one with an additional uniaxial component. There will also be DDIA guideblock that can be interchanged with the DT-25. XPD is a monochromatic beamline optimized to operate at 50 keV, but tunable from 30 to 80 keV. The system can be accessed by the standard NSLS II proposal system.

Another DDIA-30 module will be installed at Jing’s laboratory at Case Western Reserve University in the summer of 2016. This system will be used for offline testing of the cell assemblies, after initial test at APS and NSLS-II with established load versus pressure and temperature calibration curves. Such an offline system will add year-round access to the DDIA-30 module complementary to the beamline facility, and should significantly accelerate the technical development of cell assemblies.

Work Plan

We will develop experimental protocols and cell assemblies for both T-25 type and DDIA-30 type configurations, to expand pressure capability for the entire community. Major technical development tasks include designing working cell assemblies for each of the three scientific goals, optimizing anvil geometry for “ultra-high” pressures, and providing calibrations for the assemblies developed.

Tapered anvils may be required to reach ~50 GPa. At the same time, sample volume should be maximized, which means anvil TEL should be kept as large as possible. The two geometric parameters for the cell assembly, H, and T (Fig. 1), will be systematically explored to optimize designs of cell assemblies. Additional to these geometric variables are the materials variables. The “octehedra”, for example, may be made of ZrO_2, Cr- or Co-doped MgO, or other ceramic materials with low thermal conductivity. $LaCrO_3$, TiB_2, or metals may be used as heaters. Since each scientific goal has different requirements for sample size, temperature capabilities, control of oxygen fugacity, etc., a coordinated, systematic testing approach is the only way to achieve our goals. Testing grids will be established through group discussion and careful planning. PIs will be divided into three special interest groups (SIG) and be assigned to special tasks to test a given set of parameters.

Pressure and temperature capabilities will first be tested at beamlines. In the initial phase, samples may or may not be directly related to the scientific projects. After pressure-load calibration curves are reasonably established, offline tests will be performed to fine-tune the cell designs. Once a cell design reaches a stable phase, which is likely to take the first year, parts will be ordered through the COMPRES Cell Assembly Development Project.

Result Dissemination

Design and development and applications of DELVE assembly products will be published in papers and on the COMPRES Cell Assembly Development Project website. Members of the community can order these parts for them to use. At the end of this project, workshops will be organized to provide hands-on training for those interested in any of the three types of experiments.

4.3.3. Development of an Electrical Cell in the Multi-Anvil to Study Planetary Deep Interiors (PI: Anne Pommier, UC San Diego)

We propose to design, develop, and optimize a new multi-anvil sample cell that allows advances in electrical conductivity measurements. Electrical measurements in the laboratory are an excellent probe of the physics and chemistry of melts and minerals. Their use among the high-pressure community has already highlighted that they provide a better understanding of important physical and chemical processes, including the nature of the lithosphere, partial melting at ridges and subduction zones, and the thermal structure of planetary interiors. In addition, measurement of the pressure- and temperature- dependence of electrical conductivity of metals and alloys provides insight into the physics of electron transport and scattering—which also provides constraints on thermal conductivity of core materials, leading directly to insight about generation of Earth's geomagnetic dynamo and the coupled thermal history of the planet.

The objective of this project is to develop a standardized electrical conductivity cell to be made available via the MADCAP facility for use at other high-pressure laboratories. The cell design criteria is that the assemblies must be able to measure the electrical conductivity of iron alloys at high temperatures and pressures up to 25 GPa, corresponding to the core conditions of smaller terrestrial bodies such as Mercury and Mars, and to possible core formation conditions in the Earth. Our recent work developed a cell that provides high-quality electrical data up to ~8 GPa and we now propose to adapt this cell to work at higher pressure. We are requesting funds for a two-year period to design and test this high-pressure conductivity cell. These measurements are technically very challenging but our reliable and reproducible electrical measurements at lower pressure motivate us to adapt our current electrical setup to pressures up to ~25 GPa. This project will foster collaborations between the high-pressure laboratories at UC San Diego/SIO and ASU, as well as promoting interactions among the COMPRES community. It will also contribute to the training of an undergraduate student and a postdoc researcher at UC San Diego. Developing standardized electrical cells will also provide new technical and scientific directions for the existing MADCAP project and contribute to promote inter-laboratory communication through the COMPRES community.

Electrical studies are relevant to multiple disciplines: in geophysics, the augmentation of EarthScope's USArray with a magnetotelluric (MT) array has created an opportunity for development of electrical measurements in the laboratory to understand MT data in a petrological and geophysical context; in planetary sciences, electrical measurements on metals provide access to thermal conductivity, which is needed to understand the thermal history of planets. The successful and thus inspiring example of the COMPRES multi-anvil assemblies motivates having a standard multi-anvil setup for conductivity measurements designed for inter-laboratory purposes. Few laboratories have developed differing conductivity cells for the multi-anvil and the community would benefit from reaching consensus about technical challenges. We propose to standardize our existing electrical cell for the 14/8 assembly and adapt it to the 10/5 one as well as developing 3-electrode measurements.

Objective 1: Development of a standardized electrical cell
Our current electrical setup designed for the 14/8 assembly has been successfully used as part of two recent studies involving partial melting (Pommier et al., 2015a, b). Though these two studies present electrical measurements collected at pressure up to 6 GPa (and temperature >2000°C), our cell should allow work up to ~14 GPa. We propose to test our cell up to ~14 GPa, adapt it to the 10/5 assembly, and develop a 3-electrode method. Measurements at higher pressure require the use of the 10/5 assemblies, imposing smaller parts and sample dimensions than for the 14/8 assembly. Cell design and fabrication of parts will take place at ASU and electrical measurements at UCSD-SIO. The main challenges for the proposed work are:
- 2-electrode vs. 3-electrode measurements: A three-probe method needs to be employed to measure the resistance of metallic samples in order to eliminate the effect of electrodes (their resistance is a few Ohms whereas the sample's resistance will be in the order of micro-Ohms); with a 3-electrode method, resistances measured are due solely to the metallic sample itself.
- Calibration: The contribution of the electrodes to the measured electrical response needs to be negligible compared to the sample's response. To minimize this issue, blank experiments will be performed to carefully quantify on a large T range the influence of the cell on electrical results.
- Connection between furnace and electrodes: The small dimensions of the 10/5 assembly parts can cause contact between the furnace and electrodes (which would make a short-circuit). Thus, we will test other electrode configurations and materials (e.g. highly resistive cement) composing the cell to ensure optimal electrodes isolation.
- Noise on the electrical measurement: The voltage inside the furnace is a major source of noise on electrical measurements. Optimizing the quality of the electrical response may require the elaboration of a shielding system between the electrodes and the furnace.

Objective 2: Application to planetary outer cores
Electrical experiments using our new 10/5 electrical cell will be conducted at high temperature (~1600-2000°C) and high pressure on synthetic core materials representative of the Mercury core (~7-10 GPa) and Mars' outer core (~22-24 GPa). Analog core compositions will consist of iron + nickel for the two terrestrial bodies, and variable amounts of sulfur (from ~5 to 15 wt% S) will be added. The conductivity cell will be connected to a 1260 Solartron Impedance/Gain-Phase

Analyzer for electrical impedance measurements over large frequency range. Chemical analyses on retrieved samples will be performed at ASU.

Based on these electrical measurements, corresponding thermal conductivity and heat flux will be calculated for the outer cores of Mercury and Mars. Our laboratory-based values will be compared with heat flux estimates from existing numerical models to help constrain existing thermal models. Our results will also be compared with the heat flux determined from previous electrical experiments on iron. The outcomes of our project will help understand the time-evolution of magnetic field stability and dynamics at the scale of a planet and are thus critical to future numerical models of core-crystallization regime and planetary-scale convection.

5. Broader Impacts in Education and Outreach

Annual Meeting

The COMPRES annual meeting, held in June or July of each year, features keynote talks, many from prominent scientists in allied geoscience disciplines (seismology, geodynamics, geochemistry, planetary science), graduate student/postdoc presentations, facility and infrastructure reports, a poster session, a business meeting for election of standing committee membership, and a business meeting for COMPRES students and postdocs. In recent years the meeting has regularly included breakout sessions to discuss new research directions. Often a COMPRES workshop is scheduled the day prior to the meeting. For example in 2015, the “US Large Multi-Anvil Workshop” was held just before the annual meeting at Cheyenne Mountain, CO. We also schedule breakout sessions for grad students and postdocs, for example, at this year’s annual meeting in New Mexico we had the breakout session “Panel discussion for students and postdocs: Communicating Science (Grant Writing and Proposal Writing)” with panel members Bin Chen (University of Hawaii at Manoa), Lisa Danielson (Jacobs, NASA JSC), Steven Jacobsen (Northwestern University), Jie (Jackie) Li (University of Michigan), Tracy Rushmer (Macquarie University, Australia).

Figure 8. Group photo of the attendees at the 2016 COMPRES Annual Meeting in New Mexico.

The 2016 COMPRES Annual Meeting in Santa Ana Pueblo, NM attracted around 120 participants. Keynote speakers were Allen McNamara (Michigan State University), Qun Shen (Brookhaven National Laboratory), Peter Olson (John Hopkins University), Arianna Gleason (Los Alamos National Lab/Stanford University), Brandon Schmandt (University of New Mexico). We attracted nearly 60 posters and 30 of which were from students. Because it is now difficult for students to give talks at major meetings like the Fall AGU, the COMPRES Annual Meeting is commonly the place where students deliver their first professional oral presentation. COMPRES offers travel grants to the annual meeting to encourage participation by young scientists and new investigators.

Here is a list of Annual Meeting keynote speakers 2012-2015:

Frederic Bejina
Nancy Chabot
Elizabeth Cottrell
Przemyslaw Dera
Ulrich Faul
Alessandro Forte
Malcom Guthrie
Julia Hammer
Robert Hazen
Tetsuo Irifune
Jessica Irving
Zhicheng Jing
Tomo Katsura
Vedran Lekic
Robert Liebermann
Meghan Miller
Lowell Miyagi
Sujoy Mukhopadhyay
Nancy Ross
Takeshi Sakai
Carmen Sanchez-Valle
David Stegman
Tim Strobel
John Tarduno
Lara Wagner
Jessica Warren
Renata Wentzcovitch
Yao Wu

Distinguished Lecturer Program

In 2008, COMPRES established a Distinguished Lecture series in the field of mineral physics. The goal is to (1) inform students and colleagues in geosciences and related fields about how high-pressure research is contributing to the understanding of Earth processes, (2) inform interested undergraduates about research opportunities within the COMPRES community, and (3) to promote visibility for mineral physics and a better understanding of scientific problems in studies of the deep Earth. The primary target audience for these lectures is undergraduates in departments of geology and related sciences at non-PhD granting institutions, but applications from all academic institutions in the U.S. are welcome. Lecturers tailor their talks to be accessible to a broad audience at the upper-level undergraduate level. The talks feature topics that emphasize the exciting high-pressure geoscience research being conducted within the COMPRES community and its significance for understanding fundamental Earth and planetary processes.

The speakers selected for the COMPRES lecture program in COMPRES III were:
2011-2012 Abby Kavner (UCLA), Andrew Campbell (Chicago)
2012-2013 James Tyburczy (ASU), Elizabeth Cottrell (SI)
2013-2014 Jennifer Jackson (Caltech), Mark Frank (NIU)
2014-2015 Lowell Miyagi (Utah), Przemyslaw Dera (Hawaii)
2015-2016 Heather Watson (RPI), Kanani Lee (Yale)

This in this year's program Heather Watson offered two talks: "A picture is worth a million voxels: Quantitative 3-D visualization of Earth materials" and "From Comets to Cores: Unraveling the thermal history of planetary bodies". Kanani Lee offered the talks: "Figuring out the Earth from the inside out" and "Planetary Diversity: From diamond planets to hot Jupiters".

Workshops

COMPRES supports open community workshops each year that cover a variety of topics relating to science and methodologies. Workshops serve to educate users about newly developed experimental techniques and their coupled scientific applications and to broaden and strengthen user communities of COMPRES-supported facilities. For graduate students and potential new users of facilities, COMPRES offers intensive hands-on workshops designed to highlight scientific applications of COMPRES-funded resources, and to bring attendees up to speed in newly developed specific techniques. A list of workshops held in 2012-2016 is given below. These workshops were well attended, and have been successful in bringing in new users for specific techniques and capabilities, and forging and cementing scientific collaborations. Some of these workshops are offered in coordination with other organizations. Travel and workshop expenses are supported for students, post docs and young investigators based on both merit and financial need.

COMPRES-sponsored Workshops (2012-2016):

- "The 3rd FIB Training Session", February 22-23, 2012, Carnegie Institution of Washington
- "Novel Opportunities for Materials Crystallography at the ALS", October 9, 2012, Lawrence Berkeley National Laboratory
- Workshop on Data Evaluation using CONUSS & Phoenix, November 2-4, 2012, Advanced Photon Source, Argonne National Laboratory
- "Crystal Structure Prediction Workshop", December 10-15, 2012, Stony Brook University
- "Rietveld Workshop", December 8-9, 2012, Advanced Light Source, Lawrence Berkeley National Laboratory
- "7th North American Mössbauer Symposium, January 11-12, 2013, University of Texas, Austin, TX
- "The 4th FIB Training Session", February 20-23, 2013, Carnegie Institution of Washington
- "Paris-Edinburgh Cell Workshop", May 23-24, 2013, Advanced Photon Source, Argonne National Laboratory
- "Earth Cube Workshop", November 12-14, 2013, Alexandria, VA
- "MTEX Training Workshop", December 6, 2013, San Francisco, CA
- "HPCAT workshop on high-pressure time-resolved synchrotron techniques", September 25, 2014, Advanced Photon Source, Argonne National Laboratory
- "Mineral Physics Planning Workshop", October 10-12, 2014, Advanced Photon Source, Argonne National Laboratory
- "Workshop on Nuclear Resonant Scattering", November 7-9, 2014, Argonne National Laboratory

References

Bass, J., ed. Current and Future Research Directions in High-Pressure Mineral Physics. Report to the National Science Foundation. COMPRES Consortium, 28 pp. (2004).

Boulard, E., D Pan, G Galli, Z Liu, W Mao (2015) Tetrahedrally Coordinated Carbonates in Earth's Lower Mantle, Nat. Commun., 6, 6311.

Catalli, K., Shim, S-H., Prakapenka, V. B., Zhao, J., Sturhahn, W., Chow, P., Xiao, Y., Haozhe Liu, Cynn, H., Evans, W. J. (2010). Spin state of ferric iron in MgSiO3 perovskite and its effect on elastic properties. Earth Planet. Sci. Lett. 289 (1-2), 68.

Catalli, K., Shim, S-H., Dera, P., Prakapenka, V. B., Zhao, J., Sturhahn, W., Chow, P., Xiao, Y., Cynn, H., Evans, W. J. (2011). Effects of the Fe3+ spin transition on the properties of aluminous perovskite—New insights for lower-mantle seismic heterogeneities. Earth Planet. Sci. Lett. 310 (3-4), 293.

Chen, B., Z. Li, D. Zhang, J. Liu, M. Y. Hu, J. Zhao, W. Bi, E. E. Alp, Y. Xiao, P. Chow, J. Li (2014) Hidden carbon in Earth's inner core revealed by shear softening in dense Fe7C3. Proc. Nat. Acad. Sci. U.S.A., 111(50), 17755–17758, doi:10.1073/pnas.1411154111.

Chen, J., L. Li, D. J. Weidner, and M. Vaughan (2004.), Deformation Experiments using Synchrotron X-rays: In situ stress and strain measurements at high pressure and temperature, *Physics of the Earth and Planetary Interiors*, *143-144*, 347-356.

Chen, B., Jackson, J. M., Sturhahn, W., Zhang, D., Zhao, J., Wicks, J. K., Murphy, C. A. (2012). Spin crossover equation of state and sound velocities of (Mg0.65Fe0.35)O ferropericlase to 140 GPa. J. Geophys. Res. 117, B08208.

Dauphas, N., Roskosz, M., Alp, E.E., Neuville, D.R., Hu, M.Y., Sio, C.K., Tissot, F.L.H., Zhao, J., Tissandier, L., Médard, E., Cordier, C. (2014). Magma redox and structural controls on iron isotope variations in Earth's mantle and crust. Earth Planet. Sci. Lett. 398, 127.

Dera, P. and Weidner, D., eds. Mineral Physics Harnessing the Extremes: From Atoms and Bonds to Earthquakes and Plate Tectonics. Report to the National Science Foundation. COMPRES Consortium, 36 pp. (2016).

Durham, W., D. Weidner, S. Karato, and Y. Wang (2002), New developments in deformation experiments at high pressure, in *Plastic deformation of minerals and rocks*, edited by S. Karato and R. Wenk, pp. 291-329, Mineralogical Society of America, Washington D.C.

Fischer R. A., Campbell A. J., Caracas R., Reaman D. M., Heinz D. L., Dera P., and Prakapenka V. B. (2014) Equations of state in the Fe–FeSi system at high pressures and temperatures. J. Geophys. Res. 119, 2810-2827

Girard, J., Amulele, G., Farla, R. and Karato, S., (2016) Shear deformation experiment of bridgmanite + magnesiowüstite aggregates under the lower mantle conditions, Science, 351: 144-147.

Gu, T., M. Li, C. McCammon and K. K. M. Lee, Redox-induced density contrast and implications for mantle structure and primitive oxygen, Nature Geoscience, in press (2016).

Hu, Q., D.Y. Kim, W. Yang, L. Yang, Y. Meng, L. Zhang, H.-K. Mao (2016) FeO_2 and FeOOH under deep lower-mantle conditions and Earth's oxygen–hydrogen cycles. Nature, 531, 241-244.

Hunt, S. A., D. J. Weidner, R. J. McCormack, M. L. Whitaker, E. Bailey, L. Li, M. T. Vaughan, and D. P. Dobson (2014), Deformation T-Cup: A new multi-anvil apparatus for controlled strain-rate deformation experiments at pressures above 18 GPa, *Review of Scientific Instruments*, *85*(8), doi:10.1063/1.4891338.

Jackson, J. M., Sturhahn, W., Shen, G., Zhao, J., Hu, M. Y., Errandonea, D. , Bass, J. D., Fei, Y. (2005). A synchrotron Mössbauer spectroscopy study of (Mg, Fe)SiO3 perovskite up to 120 GPa. Am. Mineral. 90 (1), 199.

Jackson, J. M., Sturhahn, W., Lerche, M., Zhao, J., Toellner, T. S., Alp, E. E., Stanislav V. Sinogeikin, Jay D. Bass, Murphy, C. A., Wicks, J. K., (2013). Melting of compressed iron by monitoring atomic dynamics. Earth Planet. Sci. Lett. 362, 143.

Kono, Y., C. Kenney-Benson, D. Hummer, H. Ohfuji, C. Park, G. Shen, Y. Wang, A. Kavner, C.E. Manning (2014) Ultralow viscosity of carbonate melts at high pressures, Nature Communications, 5:5091.

Kunimoto, T., Irifune, T., Tange, Y., Wada, K., 2016. Pressure generation to 50 GPa in Kawai-type multianvil apparatus using newly developed tungsten carbide anvils. High Pres. Res. in press.

Leinenweber, K., Tyburczy, J.A., Sharp, T.G., Soignard, E., Diedrich, T., Petuskey, W.B., Wang, Y., Mosenfelder, J.L., 2012. Cell assemblies for reproducible multi-anvil experiments (the COMPRES assemblies). The American Mineralogist 97, 353-368.

Li, L., D. Weidner, P. Raterron, J. Chen, and M. Vaughan (2003), Stress measurements of deforming olivine at high pressure, *Physics of the Earth and Planetary Interior*, *in Press*.

Li, L., D. Weidner, P. Raterron, J. Chen, and M. Vaughan (2004a), Stress measurements of deforming olivine at high pressure, *Physics of the Earth and Planetary Interior*, *143-144*, 357-367.

Li, L., D. J. Weidner, J. Chen, M. T. Vaughan, M. Davis, and W. B. Durham (2004b), X-ray strain analysis at high pressure: Effect of plastic deformation in MgO, *Journal of Applied Physics*, *95*(12), 8357-8365.

Lin, J-F., Watson, H., Gyorgy Vanko, Alp, E. E., Prakapenka, V. B., Dera, P., Struzhkin, V. V., Kubo, A., Zhao, J., McCammon, C., Evans, W. J. (2008). Intermediate-spin ferrous iron in lowermost mantle post-perovskite and perovskite. Nat. Geoscience 1, 688-691.

Lin, J-F., Sturhahn, W., Zhao, J., Guoyin Shen, Mao, H.-K., Hemley, R. J. (2005). Sound velocities of hot dense iron: Birch's Law revisited. Science. 308, 1892.

Liu, J., Lin, J-F., Alatas, A., Bi, W. (2014). Sound velocities of bcc-Fe and Fe0.85Si0.15 alloy at high pressure and temperature. Phys. Earth Planet. In. 233, 24.

Liu, J., Lin, J-F., Alatas, A., M. Hu, Zhao, J., Dubrovinsky, L. (2016). Seismic parameters of hcp-Fe alloyed with Ni and Si in the Earth's inner core. J. Geophys. Res. Solid Earth, 121, 10.1002/2015JB012625.

Liu, J. (2015). The role of iron in the Earth's deep interior. Ph.D. dissertation, University of Texas at Austin.

Mao, H.-k., J. Shu, G. Shen, R. J. Hemley, B. Li, and A. K. Singh (1998), Elasticity and Rheology of Iron Above 220 GPa and the Nature of the Earth's Inner Core, *Nature*, *296*(24), 741-743.

Mao, Z., Lin, J-F., Liu, J., Alatas, A., Gao, L., Zhao, J., Mao, H.-K. (2012). Sound velocities of Fe and Fe-Si alloy in the Earth's core. Proc. Natl. Acad. Sci. USA 109 (26), 10239.

Marquardt,H., L. Miyagi (2015) Slab stagnation in the shallow lower mantle linked to an increase in mantle viscosity. Nature Geosci 8, 311–314.

Palot, M., S.D. Jacobsen, J.P. Townsend, F. Nestola, K. Marquardt, N. Miyajima, J.W. Harris, T. Stachel, C.A. McCammon, and D.G. Pearson (2016) Evidence for H2O-bearing fluids in the lower mantle from diamond inclusion. Lithos, in press, doi: 10.1016/j.lithos.2016.06.023.

Panero, W. R., J. S. Pigott, D. M. Reaman, J. E. Kabbes, Z. Liu (2015) Dry (Mg,Fe)SiO_3 perovskite in the Earth's lower mantle, JGR, 120(2), 894-908.

Schmandt, B., S.D. Jacobsen, T.W. Becker, Z. Liu, and K.G. Dueker (2014) Dehydration melting at the top of the lower mantle. Science 344, 1265-1268.

Shimomura, O., Yamaoka S., Yagi T., Wakatsuki M., Tsuji K., Kawamura H., Hamaya N., Fukuoga O., Aoki K., and Akimoto S. (1985), Multi-anvil type X-ray system for synchrotron radiation, Terra Scientific Publishing.

Singh, A. K. (1993), The lattice strains in a specimen (cubic symmetry) compressed nonhydrostatically in an opposed anvil device, *J. Appl. Phys.*, *73*, 4278-4286.

Thompson E.C., Chidester, B.A., Fischer, R.A., Myers, G.I., Heinze, D.L., Prakapenka, V.B., and Campbell A.J. (2016) Equation of state of pyrite to 80 GPa and 2400 K. American Mineralogist. 101, 1046-1051.

Wada, K., 2015. Development of new tungsten carbides and the application to high pressure apparatus, Geodynamics Research Center. Ehime University, Matsuyama.

Wang, Y., Jing, Z., Hilairet, N., Yu, T., Nishiyama, N., Tange, Y., Sakammaki, T., Rivers, M., Sutton, S., 2010. DDIA-30: a versatile megabar mutlianvil device for in-situ high pressure studies with white and monochromatic synchrotron radiation. Eos Trans. AGU, MR13A-1908.

Wicks, J. K., Jackson, J. M., Sturhahn, W. (2010). Very low sound velocities in iron-rich (Mg,Fe)O: Implications for the core-mantle boundary region. Geophys. Res. Lett. 37, L15304.

Vaughan, M., J. Chen, L. Li, D. Weidner, and B. Li (2000), Use of X-ray imaging techniques at high-pressure and temperature for strain measurements, paper presented at AIRAPT-17, Universities Press, Hyderabad, India.

Wang, Y., W. Durham, I. C. Getting, and D. Weidner (2003), The deformation-DIA: A new apparatus for high temperature triaxial deformation to pressure up to 15 GPa, *Rev. Sci. Instrum.*, *74*, 3002-3011.

Weidner, D. J., L. Li, M. Davis, and J. Chen (2004), Effect of Plasticity on Elastic Modulus Measurements, *Geophys. Res Lett.*, *31*, 6621.

Williams, Q., ed. Understanding the Building Blocks of the Planet: The Materials Science of Earth Processes. Report to the National Science Foundation. COMPRES Consortium, 68 pp. (2010).

Yamazaki, D., and S. Karato (2001), High-pressure rotational deformation apparatus to 15 GPa, *Rev. Sci. Instrum.*, *72*, 4207-4211.

Zhang, D., J.M. Jackson, J. Zhao, W. Sturhahn, E.E. Alp, M.Y. Hu, T.S. Toellner, C.A. Murphy, and V.B. Prakapenka (2016) Temperature of Earth's core constrained from melting of Fe and Fe0.9Ni0.1 at high pressures. Earth and Planetary Science Letters, 447.