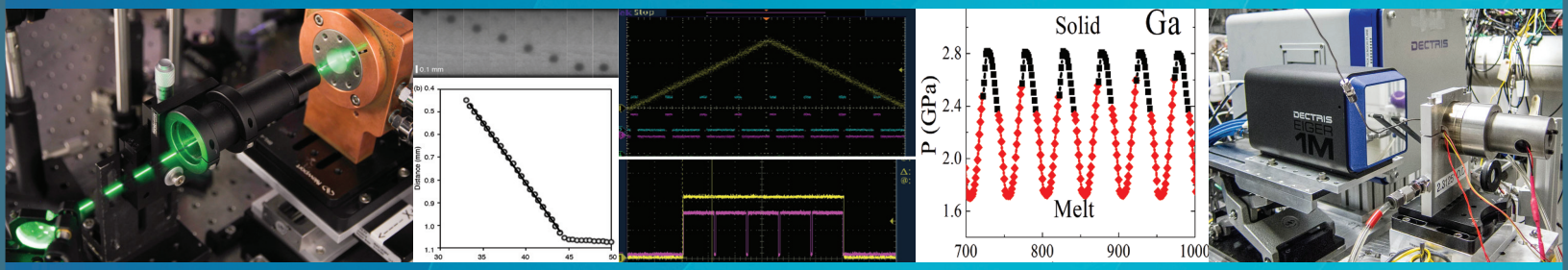

Workshop on High-Pressure Time-Resolved Synchrotron Techniques

September 25-27, 2014
Advanced Photon Source,
Argonne National Laboratory,
Argonne, IL U.S.A.

Sponsors:

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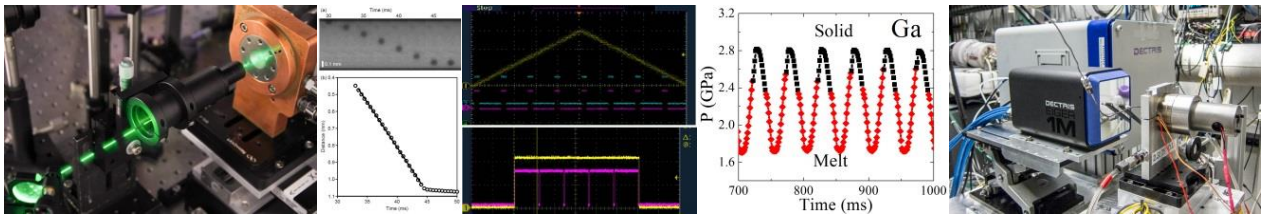
HPCAT HIGH PRESSURE COLLABORATIVE ACCESS TEAM
at the Advanced Photon Source
GEOPHYSICAL LABORATORY, Carnegie Institution of Washington



Welcome to the Workshop on high-pressure time-resolved synchrotron techniques 2014

Complementary advances in synchrotron sources, x-ray optics, fast area detectors, IR lasers and adaptable remote pressure controls have recently made possible many time-resolved experimental techniques for studying materials at extreme pressure and temperature conditions. The High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source has made a sustained effort over the past several years to assemble a powerful collection of high pressure apparatus aimed at time-resolved studies of materials, and considerable time has been invested in developing techniques for collecting high-quality time-resolved x-ray scattering data.

Techniques currently available, or under development, include (but are not limited to): pulsed/ramp laser-heating for thermal equation of state and melting experiments; rapid, controlled uni-directional or cyclical sample compression/decompression—with rates ranging from less than a GPa per second to extreme compression in excess of a TPa per second—for synthesis/observation of metastable high pressure phases, phase transition kinetics and fast rheology; high speed imaging for measuring viscosity at high pressure and temperature; and Laue (white beam) measurements to study phase transitions as they unfold.

Given these recently emerging technology developments at HPCAT and other synchrotron and dynamic experimental facilities, it is an opportune time for the static high pressure community to come together and exchange ideas, report first results, and discuss future plans. Thus, HPCAT is presenting a two and half day workshop dedicated to time-resolved x-ray scattering techniques.

One of the purposes of the workshop is to exchange ideas through invited and contributed talks, discussion periods, and an evening poster session. The workshop is attended by a diverse group of researchers in high pressure and time resolved techniques, with a significant number of young researchers, and we anticipate the outcome will have a significant impact in understanding matter at extreme conditions.

Workshop Purpose and Goals

- Introduce time-resolved apparatus and techniques currently available at HPCAT and other synchrotron facilities
- Present the results of time-resolved high pressure research carried out at HPCAT and other facilities
- Motivate students and early career researchers to pursue time-resolved research programs
- Demonstrate available facilities and software during hands-on practical sessions
- Identify scientific goals and associated technical challenges for future time-resolved studies
- Anticipate the expanded scope of time-resolved high-pressure research that will be enabled by the proposed APS MBA upgrade

Workshop Organizing Committee

- Stanislav Sinogeikin, *HPCAT / Carnegie Institution of Washington*
- Thomas Duffy, *Princeton University*
- Will Evans, *Lawrence Livermore National Laboratory*
- John Tse, *University of Saskatchewan, Saskatoon, Canada*
- Nenad Velisavljevic, *Los Alamos National Laboratory*
- Choong-Shik Yoo, *Washington State University*

Local organizing Committee (HPCAT/CIW)

Stanislav Sinogeikin, Yoshio Kono, Yue Meng, Dmitry Popov, Guoyin Shen, Jesse Smith, Yuming Xiao

Local Administrator

Freda Humble, (630) 252-0479, *fhumble@ciw.edu*

Workshop contact

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Workshop on high-pressure time-resolved synchrotron techniques

September 25-27, 2014, Advanced Photon Source

Comprehensive Program

Workshop on high-pressure time-resolved synchrotron techniques

September 25-27, 2014, Advanced Photon Source

Comprehensive Program

Wednesday, September 24, 2014

17:00 **Registration and Reception (cash bar + finger food; concludes at 19:30) –** *Argonne Guest House*

Thursday, September 25, 2014

7:30 **Poster setup –** *in the Gallery, lower level APS Conference Center (Building 402)*

7:30 **Registration, Continental Breakfast –** *APS Auditorium (Building 402)*

Note: **All talks will be held in APS Auditorium**

Session 1: Introduction

Chair: Guoyin Shen

8:30 **Welcome,** *Stas Sinogeikin (HPCAT)*

8:40 **Overview of HPCAT and future perspectives for time-resolved experiments,** *Ho-Kwang (Dave) Mao (HPCAT, CIW)*

9:10 **MBA upgrade and effect on time-resolved experiments,** *Dennis Mills (APS)*

9:40 **Coffee break**

Session 2: Time-resolved high-pressure x-ray imaging

Chair: Nenad Velisavljevic

10:00 **High-pressure x-ray imaging experiments at HPCAT,** *Yoshio Kono (HPCAT)*

10:25 **Study of liquid-liquid immiscibility (silicate melt-water) by x-ray imaging in large volume press,** *Kenji Mibe (U. Tokyo)*

10:50 **X-ray fast tomography and its applications in dynamical phenomena studies in geoscience at APS,** *Xianghui Xiao (APS)*

11:15 **High-pressure and high-speed liquid jets: shock waves, cavitations and dynamics,** *Jin Wang (APS)*

11:40 **Discussion**

11:50 **Group photo -** *Front steps*

12:00 **Lunch -** *Gallery, lower level APS Conference Center (Building 402)*

Session 3: X-ray micro-diffraction with fast compression and decompression

Chairs: Choong-Shik Yoo and Jesse Smith

1:00 **Fast (de)compression capabilities and first experimental results at HPCAT,** *Jesse Smith (HPCAT)*

1:25 **Time-resolved X-ray diffraction and electrical resistance measurements on metals at high pressure,** *Nenad Velisavljevic (LANL)*

1:50 **Rate dependent pathways for the high pressure synthesis of novel metastable phases in Group IV elements,** *Bianca Haberl (ANU/ORNL)*

2:15 **Dynamic response of materials by oscillatory deformation experiment + X-ray imaging,** *Don Weidner (Stony Brook)*

2:40 **Discussion**

2:50 **Coffee break**

3:10 **Modeling phase transition kinetics under dynamic compression,** *Carl Greeff (LANL)*

3:35 **Kinetics of structural phase transitions and fast x-ray diffraction,** *John Tse (USASK)*

4:00 **X-ray transient absorption spectroscopy for solar energy research,** *Lin Chen (NU/ANL)*

4:25 **Challenges and opportunities in analysis of massive data from time resolved experiments,** *Przemek Dera (UH)*

4:50 **Discussion**

5:00 **Poster session** – *in the Gallery, lower level APS Conference Center (Building 402)*
Chair: Stanislav Sinogeikin

6:30 **Workshop Dinner –** *Gallery, lower level APS Conference Center (Building 402)*

Friday, September 26, 2014

7:30 **Continental Breakfast –** *APS Conference Center (Building 402)*

Session 4: Pulsed/ramped laser heating and x-ray micro-diffraction

Chairs: Tom Duffy and Yue Meng

8:30 **High PT experiments with pulsed laser heating: capabilities at HPCAT,** *Yue Meng (HPCAT)*

8:55 **Technical developments on high-pressure melting of metals using pulsed laser heating,** *Ross Hrubiak (HPCAT)*

9:20 **Time-resolved x-ray diffraction of solids under dynamic loadings,** *Choong-Shik Yoo (WSU)*

9:45 **Melting measurements using XAS at megabar pressures,** *Reini Boehler (CIW)*

10:10 **Discussion**

10:20 **Coffee break**

10:40 **Time-domain experiments at extreme conditions at GSECARS,** *Vitali Prakapenka (GSECARS)*

11:05 **Single-pulse laser heating in diamond anvil cells: Probing material under planetary interior conditions,** *Alex Goncharov (CIW)*

11:30 **Fast temperature readout spectrometer for atomic dynamics measurement,** *Dongzhou Zhang (APS/Caltech)*

11:55 **Time-resolved X-ray diffraction at APS beamline 7ID,** *Don Walko (APS)*

12:20 **Discussion**

12:30 **Lunch -** *Gallery, lower level APS Conference Center (Building 402)*

Session 5: New time-resolved high-pressure X-ray techniques and directions and related topics

Chairs: John Tse and Yuming Xiao

1:30 **Laue capabilities at HPCAT - towards time-resolved studies,** *Dmitry Popov (HPCAT)*

1:55 **Dynamic Laue diffraction: A probe for understanding the kinetics of strength and phase transitions,** *Joel Bernier (LLNL)*

2:20 **100 ps time-resolved white beam diffraction at BioCARS,** *Keith Moffat (UC)*

2:45 **Time-resolved XES,** *Tsu-Chien Weng (SSRL)*

3:10 **Discussion**

3:20 **Coffee break**

3:40 **Fast detectors for time resolved studies – current status and new developments,** *Robert Bradford (APS)*

4:05 **Laser-based dynamic compression of solids,** *Tom Duffy (Princeton)*

4:30 **Ultrafast x-ray scattering measurements from dynamically compressed solids,** *Siegfried Glenzer (LCLS)*

4:55 **A first laser shock experiment at the ESRF to probe Warm Dense Iron,** *Florent Occelli (DPTA/SPMC)*

5:20 **Discussion**

5:30 **Posters / free time** (*take down posters between 6-6:30*)

6:30 **Workshop Dinner –** *Gallery, lower level APS Conference Center (Building 402)*

Saturday, September 27, 2014

8:00 **Continental Breakfast –** *HPCAT / Building 434C*

Parallel hands-on / demonstration sessions

NOTE: ***Four parallel hands-on / demonstrations sessions will be held simultaneously at four different stations. Attendees can sign up for groups (A, B, C, or D) during registration.***

	Fast de/compression and cyclic pressure generation and control in DAC, *Stas Sinogeikin and Chuanlong Lin*	**Pulsed and ramped laser heating in DAC,** *Yue Meng and Ross Hrubiak*	**Fast imaging,** *Yoshio Kono and Curtis Kenny-Benson*	**Large-volume data reduction and analysis,** *Przemek Dera, Jesse Smith and Clemens Prescher*
	16-IDB area	**16-IDB**	**16-BMB**	**434C Conf. room**
9:00	**A**	**B**	**C**	**D**
9:35	**D**	**A**	**B**	**C**
10:10	**C**	**D**	**A**	**B**
10:45	**B**	**C**	**D**	**A**

11:20 **Tour of HPCAT facilities (same 4 groups)**

12:00 **Lunch –** *HPCAT / Building 434C*

13:00 **Adjourn**

Abstracts:
Oral presentations

1.1. Overview of HPCAT and future perspectives for time-resolved experiments

Ho-kwang (Dave) Mao

HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Washington DC, USA

HPCAT at Sector 16 of the Advanced Photon Sources (APS) is dedicated to the advancement of high-pressure sciences. A large battery of synchrotron x-ray diffraction, spectroscopy, and imaging techniques are integrated at two canted undulator and two bending-magnet beamlines of the HPCAT for in-situ high pressure-temperature (P-T) investigations of materials behaviors with x-ray probes of high spatial, energy, and momentum resolutions. Recently the importance of the time (t) variable has been widely recognized with the great progress in temporal resolutions of experimental control and probe-detector capability. Major impacts are emerging in the following high-pressure research areas:

(1) Mass transport under high *P-T*: elemental diffusion, viscosity, high strain rate, dynamic response.

(2) Transient phenomena: Phase transition kinetic and mechanism, intermediate states, unstable sample conditions.

(3) Optical pump-probe investigation of materials properties at extreme *P-T*.

(4) Extended *P-T* ranges beyond the static conditions.

To fully explore the *t-P-T* dimensions, HPCAT is planning its development in coordination with the APS MBA upgrade, the advancement of instrumentation, and the availability of the next-generation free-electron laser.

1.2. MBA Upgrade and effect on time-resolved experiments

Dennis Mills

Deputy Director, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

The APS Upgrade team is developing a plan for the implementation of a multi-bend achromat (MBA) lattice into the APS as part of our upgrade plans. An MBA lattice will produce a much lower electron beam emittance in the new storage ring, resulting in an increase in x-ray beam brightness 100 to 1000 times larger than can be generated with the current storage ring lattice. A new MBA lattice will require several changes to the existing APS parameters, including electron beam energy, fill patterns, etc. These changes will be discussed in terms of their effect on future time-resolved experiments at the APS.

2.1. High-pressure x-ray imaging experiments at HPCAT

Yoshio Kono[1], Curtis Kenney-Benson[1], Changyong Park[1], Guoyin Shen[1], and Yanbin Wang[2]

[1] HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL, USA
[2] GeoSoilEnviroCARS, Center for Advanced Radiation Sources, The University of Chicago

Techniques for studying structure, properties, and behavior of liquids at high pressures have been integrated using a Paris-Edinburgh (PE) cell at the beamline 16-BM-B, HPCAT of the Advanced Photon Source (e.g., Kono et al., 2014). X-ray imaging combined with PE cell is one of the most powerful tools to investigate behavior and properties of liquids at high pressure and high temperature conditions. We utilized white X-ray phase contrast imaging for studying phase separation and immiscibility of liquids not only by density contrast but also by phase contrast image for low density contrast liquids. We succeeded to monitor immiscibility of silicate and carbonate melts, having low density contrast. In addition, we have installed high speed camera for ultrafast imaging to investigate dynamics of liquids at high pressures and high temperatures. The setup is capable of imaging up to ~100,000 frames/second (fps) in air (and probably in diamond anvil cell) and up to ~10,000 fps in the PE cell. We currently employ the ultrafast imaging technique for studying viscosity of liquids at high pressure and high temperature conditions. The ultrafast imaging enabled us to overcome limitation of previous viscosity measurement for low viscous liquids, and to investigate viscosity of very low viscous liquids such as liquid salts (Kono et al., 2013), which have viscosity similar to that of water. These high-pressure imaging experiments promote understanding of property and behavior of liquids at high pressures and high temperatures. In addition, these imaging experiments can be combined with liquid structure measurement. The integrated capability opens a new way to investigate the direct link between microscopic structure and macroscopic physical properties of liquids at high pressure and high temperature conditions.

References:

Kono, Y., Kenney-Benson, C., Park, C., Shen, G. & Wang, Y. Anomaly in the viscosity of liquid KCl at high pressures. *Phys. Rev. B* **87**, 024302 (2013).

Kono, Y., Park, C., Kenney-Benson, C., Shen, G. & Wang, Y. Toward comprehensive studies of liquids at high pressures and high temperatures: Combined structure, elastic wave velocity, and viscosity measurements in the Paris–Edinburgh cell. *Phys. Earth Planet. Inter.* **228**, 269-280 (2014).

2.2. Study of liquid-liquid immiscibility (silicate melt – water) by x-ray imaging in large volume press

Kenji Mibe

University of Tokyo

In the Earth's interior, it is possible that two or more fluids coexist under high pressure and temperature conditions. Those coexisting fluids might have played important role in the geochemical evolution and stratification in the Earth's interior. This is because physical and chemical properties of coexisting fluids could be different from each other and hence could move separately. In order to determine the stability fields of immiscible fluids, we have been using high pressure and temperature x-ray radiography.

Experiments were done using X-ray radiography technique together with Kawai-type multi-anvil press (SPEED-1500) installed at BL04B1, SPring-8, Japan. Direct white X-ray beam, which passes through the anvil gaps of SPEED-1500 and sample under high pressure, is observed with an X-ray camera. Real time radiographic movies can be recorded. The spatial resolution of the sample image is about 5 micrometers. The exposure time for the X-ray camera is about 35 msec.

By now, we have succeeded in experiments with some silicate-H_2O systems (Mibe et al., 2004; 2007; 2011; Kawamoto et al., 2012). The same technique was also applied to the system Fe-S-Si in order to understand the properties of Earth's core (Morard et al., 2008). Very recently, we are also trying to determine the chemical compositions of coexisting fluids by synchrotron radiation x-ray fluorescence (XRF) analysis using the same experimental configurations as the radiography (Kawamoto et al., 2013; 2014).

References:

Kawamoto et al. 2012, *Proc Nat Acad Sci USA* **109**, 18695.
Kawamoto et al. 2013, Abstract, Goldschmidt Conference.
Kawamoto et al. 2014, Abstract, 21st General Meeting of IMA South Africa 2014.
Mibe et al. 2004 *Geochim Cosmochim Acta* **68**, 5189.
Mibe et al. 2007, *J Geophys Res* **112**:B03201, 10.1029/2005JB004125.
Mibe et al. 2011, *Proc Nat Acad Sci USA* **108**, 8177.
Morard et al., 2008, *J Geophys Res* **113**, B10205, doi:10.1029/2008JB005663.

2.3. X-ray fast tomography and its applications in dynamical phenomena studies in geoscience at APS

Xianghui Xiao

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

Synchrotron radiation based fast tomography can provide high spatial and temporal resolutions that is suitable in dynamical phenomena studies in geoscience. This presentation aims at reviewing the potential of Synchrotron radiation based micro-tomography for research applications in structural geology and experimental rock mechanics. Besides outlining the technical capabilities of the latest generation of microtomography beam lines at the Advanced Photon Source (USA), we will present our workflow for the analysis of large time-resolved tomographic datasets. We will conclude with an outlook on a next generation of in-situ studies on fluid-rock interaction and rock deformation.

2.4. High-pressure and high-speed liquid jets: Shock waves, cavitations and dynamics

Jin Wang

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

High-pressure, high-speed sprays are an essential technology for many applications, including fuel injection systems, thermal and plasma spray coating, and liquid-jet machining. In the case of fuel injection, an understanding of the structure and dynamics of the fuel sprays is critical to optimizing the injection process to increase fuel efficiency and reduce pollutants. There are many intriguing dynamic effects associated with the multiphase phenomena such as shock waves and cavitations as liquid is injected at a few thousand bars. Multiphase liquid jets are also often difficult to study optically because of intense multiple light scattering from surrounding liquid droplets. We used ultrafast x-radiography and developed a novel multiphase numerical simulation to reveal the origin and the unique dynamics of the liquid-jet-generated shock waves and internal cavitations. The multiphase simulation revealed that the aerodynamic interaction between the liquid jet and the shock waves results in an intriguing ambient gas distribution in the vicinity of the shock front, as validated by the ultrafast x-radiography measurements. On the other hand, we demonstrated that a slight alteration of the geometry on the micrometer scale can induce distinct laminar-like or cavitating flows. By taking advantage of the pulsed structure of x-rays at the APS, we now use an x-ray-based velocimetry technique to characterize the near-nozzle dynamics of the supersonic diesel sprays by multi-exposed x-ray phase-contrast imaging. Notable features of the jet and spray dynamics in the near-nozzle region are discussed.

3.1. Fast (de)compression capabilities and first experimental results at HPCAT

Jesse Smith, Chuanlong Lin, Eric Rod, Stanislav Sinogeikin, and Guoyin Shen

HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439, USA

HPCAT's general purpose x-ray microdiffraction endstation is one of only a few beamlines in the world that combines a high-energy 3rd generation synchrotron source, high-throughput x-ray optics, remote and precise sample pressure control, and high-frequency x-ray area detectors. These elements, together with freely available software capable of automated processing of large volumes of data, make possible a number of high-pressure experimental techniques based on the rapid compression and/or decompression of materials in the diamond anvil cell.

The various pressure control apparatus offer a number of options for the time-dependent sample pressure profile. I will present several examples highlighting some of the pressure profile options, including rapid and continuous pressure increase for equation of state measurements at ambient and elevated temperatures, rapid sample decompression for synthesis of amorphous materials, ultrafast compression to generate high strain rates, and cyclic compression/decompression to observe lattice relaxation in materials. Finally, some current challenges and possible future directions in fast (de)compression experiments are discussed.

3.2. Time-resolved X-Ray diffraction and electrical resistance measurements on metals at high P-T

Nenad Velisavljevic

Shock and Detonation Physics group, Los Alamos National Laboratory, Los Alamos, NM 87545

We have tested a new experimental capability for achieving high pressure at variable compression rate with standard gas-membrane Diamond Anvil Cell (DAC). The new capability utilizes a gas buffer and a solenoid valve for controlled gas delivery to the membrane. Various aspects of gas controller, as well as the results of some of the test measurements performed will be discussed. In particular, test measurements and initial high pressure variable compression rate work was performed on zirconium (Zr) and other group IVa metals. During preliminary experiments pressure jumps of 80 GPa or higher were systematically obtained in less than 0.2s (~400GPa/s). X-ray diffraction and electrical resistance measurements were used to provide first time resolved data on α → ω → β structural evolution in Zr during this rapid pressure increase. Direct control of pressure increase and compression rates using both the gas-membrane controller and dDACs with piezoelectric actuator [1] allows for investigation of structural evolution and kinetics of structural phase transitions of materials under previously unexplored compression rate - pressure conditions that bridge traditional static and shock/dynamic experimental platforms.

Reference:

[1] W. J. Evans, et al., *Rev. Sci. Inst*. **78**, 073904

3.3. Rate dependent pathways for the high pressure synthesis of novel metastable phases in Group IV elements

Bianca Haberl[1,*], Malcolm Guthrie[2,], Jesse S. Smith[3], Stanislav V. Sinogeikin[3], Guoyin Shen[3], Jim S. Williams[1], and Jodie E. Bradby[1]**

[1]Department of Electronic Materials Engineering, Research School of Physics and Engineering, Australian National University, Canberra, ACT 0200, Australia
[2]Geophysical Laboratory, Carnegie Institution of Washington, Washington DC 20015, USA
[3]High Pressure Collaborative Access Team, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439, USA

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The elemental semiconductor silicon, but also germanium, in the standard diamond-cubic phase form the basis of our current semiconductor technology. Interestingly, both elements exhibit pressure-induced polymorphism where strong kinetic barriers allow for the synthesis of crystalline metastable phases. These exhibit significantly altered band gap characteristics, better overlap with the solar spectrum or different resistance to etching. Thus their formation could be exploited for improvement of existing devices through pressure-induced band-gap engineering of Si and Ge itself.

To potentially utilize these phases industrially, point loading as mode of pressure application is advantageous. In contrast to synthesis in a diamond-anvil cell, it enables formation of these structures incorporated into the surface of standard wafers as used in existing semiconductor industry. Furthermore, such point loading also allows for precise control of decompression rates, one of the key parameters controlling the formation of metastable phases. For targeted synthesis, however, a full understanding of the transition pathways is essential. This cannot be achieved by point loading, but only by in situ observation in a diamond anvil cell. Therefore, control over decompression rates in a diamond-anvil cell together with the necessary collection of high-quality time-resolved X-ray diffraction data are necessary to fully explore the formation of these interesting metastable structures of Si and Ge.

Clearly, this has become possible through the development of time-resolved synchrotron techniques at HPCAT and completely new insights have been gained. For example, for Ge no strong dependence on the decompression rate is observed, contrary to previous reports. For Si, however, phase formation upon decompression is critically rate dependent with fast decompression yielding an amorphous and slow decompression a body-centered cubic (bc8) phase. Both these structures are synthesized from metallic Si polymorphs stable between ~10-15 GPa. However, preliminary data suggest that rapid decompression from different metallic structures, formed at low temperatures or higher pressures, can yield a number of additional interesting Si polymorphs. Thus further exploration of the dependence of the polymorphism of these Group IV elements on decompression rates clearly opens the scope for the synthesis of further, potentially useful structures.

3.4. Dynamic response of materials by oscillatory deformation experiment + x-ray imaging

Donald J. Weidner

Stony Brook University

The deformation-DIA (D-DIA) at the COMPRES beamline at the NSLS has been a fruitful apparatus for defining dynamic properties of materials at pressure to 10 GPa and temperatures to 2000K. The DDIA is a high pressure tooling that accommodates samples with a few mm dimensions and can apply a uniaxial stress superimposed on a hydrostatic pressure. The stress can be applied constantly over several hours or oscillatory with a frequency ranging from 1 Hz to 1 μHz. The x-ray diffraction signal can be used to define the magnitude of stress and images can be used to define strain. Images of sample and a reference standard can also be used to define both the strain and the stress field.

Here we present examples of oscillations fields of both stress and temperature that have been used to define rheological properties, elastic properties, elastic attenuation (Q), phase transformations, kinetics of phase transformations, and thermal diffusivity.

3.5. Modeling phase transition kinetics under dynamic compression

Carl W. Greeff

T-1, Los Alamos National Laboratory

I will discuss the use of static and dynamic compression data together with first principles calculations in developing multi-phase equations of state. Time-resolved dynamic compression data, both shock and ramp loading, contain a wealth of information on phase transitions. The interpretation of this data often involves simulations with kinetic models. These are typically formulated as homogenized continuum models in which the phase fractions evolve at rates that are functions of Gibbs free energies. I'll describe a particular model of this type, which emphasizes the strong nonlinearity of these functions. I will show applications to Zr and Sn. Finally, I will discuss a path to improving the model by implicitly including meso-scale information.

3.6. Kinetics of structural phase transitions and fast x-ray diffraction

John S. Tse

Department of Physics and Engineering Physics, University of Saskatchewan, Saskatoon, Canada

The current availability of x-ray detector with very fast cycle rate (greater than 1000 Hz) has opened up the opportunity for following a structural transformation in real time by diffraction method. Structural phase transition involves the collective displacement and/or diffusion of atoms. These processes often happen within the frequency of a lattice vibration. There are existing techniques applicable within the millisecond time scale. To fully utilize the new capability, a challenge is to choose appropriate systems with pressure-induced transformation kinetics commensurate with the time regime. Therefore the knowledge of the factors in driving a structural transformation is crucial. For this purpose, the current status on the understanding of the kinetics of pressure-induced structural transformation will be discussed. A few systems will be suggested for possible experiments.

3.7. X-ray transient absorption spectroscopy for solar energy research

Lin X. Chen

Chemical Sciences and Engineering Division, Argonne National Laboratory, Building 200, 9700 South Cass Avenue, Argonne, Illinois 60439; Department of Chemistry, Northwestern University, 2145 Sheridan Road, Evanston, Illinois 60208
lchen@anl.gov, or l-chen@northwestern.edu

Fundamental processes in solar energy conversion involve photon-matter interactions through light harvesting, excited state formation and subsequent transformation to convert light energy to electricity, heat and fuels. Intense pulsed x-ray sources from synchrotrons and x-ray free electrons lasers coupled with ultrafast lasers open up a new opportunity to reveal direct structural information of transient species in solar energy conversion processes, such as photocatalysts, photosensitizers, and photovoltaic materials. In particular, transient oxidation states of metal centers in electron donors and catalysts can be unambiguously identified with accompanying nuclear geometric transformations. X-ray transient absorption spectroscopy enables molecular snapshots after the photoexcitation, which will enable the make of molecular movies and have a tremendous impact on our understanding of the coupling between the electron transfer events and structural control parameters of participating partners in solar fuel and solar electricity generation. The lecture describes XTA method developments, the examples of its applications in solar energy research and the challenges and perspective.

3.8. Challenges and opportunities in analysis of massive data from time resolved experiments

Przemyslaw Dera

Hawaii Institute of Geophysics and Planetology, School of Ocean and Earth Science and Technology, University of Hawaii at Manoa

Synchrotron radiation user facilities are critical resources, which enable state of the art research and student training. Access to these facilities is very competitive, and time allocated for experiments always very constrained. Conventional diffraction experiments (carried out at ambient conditions with good quality samples, typically of only one crystalline phase) usually produce data that can be analyzed with a combination of the currently available software. However, the situation is much more difficult with unconventional data, e.g. produced during experiments at non-ambient conditions, or exploring the real time progress of chemical or physical processes. The unconventional experiments are extremely valuable in lab-scale modeling of technological (e.g. CO_2 sequestration, new hydrogen storage material discovery, gas separation), as well as geological processes (e.g. deformation and phase transitions of mantle minerals), but introduce significant complexities into the data, often beyond the capabilities of available analysis software.

Technology used in synchrotron experiments evolves rapidly increasing the speed of the data collection and producing massive volumes of experimental data, posing new serious challenges for data analysis. The experiments are often decision-driven, and require at least partial real time data interpretation to guide the experimenter (e.g. monitoring a progress of sample transformation, or detecting a subtle discontinuity which may require a more careful investigation). Software offering such real time analysis capabilities is currently not available.

ATREX (Advanced Tools for Research in Extreme Xtallography) is a new NSF-funded project in collaboration between University of Hawaii and Stony Brook University. Utilizing a combination of existing, well tested and widely used software components we are aiming to create a new integrated multiplatform, open-source Python software package, with unique capabilities to process diffraction image data from samples in all forms from glasses and melts, bulk powders, though coarse multi grains, to single crystals, which will support new data types produced by novel ultrafast X-ray imaging detectors, offer extensive automated serial processing capabilities for massive data sets and will allow real time data analysis for time-constrained decision-driven synchrotron experiments. ATREX will include database access capabilities utilizing the free American Mineralogist Crystal Structure Database.

4.1. High PT experiments with pulsed laser heating: capabilities at HPCAT

Yue Meng

HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439, USA

In the past two decades, laser-heated DAC coupled with in-situ XR techniques has evolved into a unique and powerful experimental tool for studying a broad range of material properties under extreme conditions. With applications of continuous-wave (CW) heating mainly to overcoming kinetic barriers to pressure-induced phase transitions and material synthesis, the development of experimental techniques in this area, accordingly, have been focusing on long term system stability and experiment probes in time scale ranging from seconds to hours. Motivated by addressing issues in experimental research of high-pressure melting and thermal equations of state, HPCAT has developed single-pulsed laser heating synchronized with temperature and XRD measurements, and established capabilities that are critical to heating quality and to reliable measurements under high PT conditions in both pulsed and CW heating experiments.

In this presentation, the following areas will be discussed:

(1) Why do we need pulsed laser heating in high PT research

(2) Current technologies – laser, XR and heating temperature detection in increasingly reduced timescales

(3) HPCAT capabilities – established and near future plan

4.2. Technical developments on high-pressure melting of metals using pulsed laser heating

Ross Hrubiak, Yue Meng, and Guoyin Shen

HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439, USA

The main purpose of this talk is to discuss several important experimental developments (brought forward by researchers in the larger high pressure community as well as at HPCAT) relevant to carrying out careful structural measurements on pure metals close to and above their melting temperature with pulse laser heated diamond anvil cell and synchronized *in-situ* x-ray diffraction.

Precise high temperature generation and measurement for many metals close to their melting temperatures is not feasible with a time-continuous laser heating in a diamond anvil cell approach due to the chaotic nature of the temperature response of the sample as well as chemical reactions which can take place over even a relatively short time scale. However, provided that a number of important experimental considerations have been met, metal samples in a diamond anvil cell can now be controllably laser heated to 6000 K for relatively long pulse periods of 10-30 milliseconds, which is enough time to simultaneously collect x-ray diffraction in a single pulse with the currently available x-ray photon flux at the beamline 16-IDB of Advanced Photon Source.

Time-scale dependent temperature response of the sample during the laser pulse duration is one of the most critical experimental variables and this talk will stress on several key strategies for attaining a desired temperature response. Several developments in areas such as sample preparation, sample chemistry during laser heating, laser pulse time-structure, alignment and synchronization of laser pulse, the temperature measurement and the x-ray beam will be discussed.

I will also present an example of an experiment involving controlled heating and measurement of x-ray diffraction of molybdenum metal samples under high pressure to temperatures thousands degrees above the melting point.

I acknowledge the collaboration with Reini Boehler and Amol Karandikar who shared their expertise on diamond anvil cell sample preparation and loading; and Vitali Prakapenka and Clemens Prescher for their help with time resolved temperature measurements.

I also acknowledge the support of DOE-NNSA, DOE-BES and NSF

4.3. Time-resolved X-ray diffraction of solids under dynamic loadings

Choong-Shik Yoo

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We present novel time-resolved (TR) x-ray diffraction and TR-Raman spectroscopy capable of probing structural and chemical evolutions of solids undergoing chemical and phase transformations. These methods are applicable to a wide range of dynamic experiments to study both single event phenomena of solids under thermal, electric or mechanical impact conditions and non-single event changes using dynamic-DAC and high frequency pulse (or ramp) laser-heated DAC. In this talk, relevant technology developments are described with several examples of our recent studies on reactive metals and dense molecular systems, which are synergistic to many proposed activities to develop dynamic synchrotron x-ray diffraction capabilities, centered at advanced third and fourth-generation light sources.

* The present study was performed in support of NSF-DMR (Grant No. 1203834), ARO (Grant # W911NF-14-1-0233) and CDAC/NNSA.

4.4. Melting measurements using XAS at megabar pressures

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Temperature, thermal history and dynamics of the Earth, rely critically on the knowledge of the melting temperature of iron at the pressure conditions of the inner core boundary (ICB) (330 GPa) where the geotherm crosses the melting curve. The amount of literature on this subject is overwhelming, but no consensus has been reached. The two experimental techniques capable of reaching pressure and temperature conditions of the Earth's core, shock compression and laser-heated diamond cells show very large disagreement ranging from 4800 K to 7600 K for the ICB temperature with similar diversity in theoretical estimates. Here we report new data on the melting temperature of iron in the laser-heated diamond cell to 103 GPa obtained by X-ray absorption spectroscopy (XAS), a technique rarely employed at such conditions. The melting criterion using XAS combined with a new sample encapsulation technique is unambiguous and highly reproducible and the results support a flat melting curve of iron with a melting temperature at the ICB of 5000 K.

4.5. Time-domain experiments at extreme conditions at GSECARS

Vitali B. Prakapenka

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The dynamic x-ray probe is the ideal choice for real time applications in static and dynamic high pressure and temperature experiments for studying physical and chemical properties of materials in Mbar pressure range when combination of the time resolved synchrotron techniques including diffraction, emission, absorption and inelastic scattering with pulse laser heating and time resolved optical diagnostic methods could be accomplished.

Recent developments in continues and pulse laser heating technique, including application of fiber lasers and flat top laser beam shaping optics, result in significant improvement of the quality of x-ray data collected in-situ at high pressures and high temperatures in the diamond anvil cell (DAC) [1]. At GSECARS (Sector 13, Advanced Photon Source), we have combined the in-situ time-resolved synchrotron x-ray diffraction technique with pulse double-sided laser heating system [2]. A frequency-modulated laser beam was synchronized with x-ray detector, temperature measuring spectrometer and APS storage ring bunch structure. Controlling delay time between x-ray pulses (500 ns for hybrid fill) and laser pulses (microseconds), e.g. measuring sample at different temperatures, we have observed the clear shift of sharp x-ray diffraction lines due to thermal expansion of the laser heated samples. Furthermore, we were be able to detect diffuse x-ray scattering from molten materials in the pulse laser heated DAC that allows us precisely constrain the high pressure melting curve in the Mbar pressure range.

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4.6. Single-pulse laser heating in Diamond Anvil Cells: Probing material under planetary interior conditions

Alexander Goncharov

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Co-authors: R.S. McWilliams, Z. Konopkova, V.B. Prakapenka, S.S. Lobanov, N. Holtgrewe

With the new high-brilliance x-ray sources, existing (e.g., LCLS) and coming online soon (e.g., EXFEL), new possibilities of investigating matter at conditions approaching warm dense matter state are emerging. New technology will help in furthering knowledge of the states and properties of matter in interiors of planets which will impact understanding of the planetary composition and structure and also planetary history. Here I will address the needs of developing techniques for creating and probing extreme pressure-temperature (P-T) conditions that would be compatible with synchrotron X-ray probes.

The main focus is to determine the state, crystal and chemical structure, and thermochemical properties of materials under previously unattainable pressure-temperature conditions using static compression technique. Pressure has been generated in conventional diamond anvil cells (up to 200 GPa); the laser power is coupled to sample through thin metallic pieces positioned in the high-pressure cavity. Pulsed-laser heating with microsecond to second's pulse duration has been utilized to reach temperatures of 2,000-15,000 K. To avoid extensive unwanted chemical reactivity and material diffusion the experiments are conducted in a single (or as small as possible number) event(s) during which material properties are measured and P-T are determined simultaneously. The duration of the experiment (few microseconds to several seconds) is chosen to collect sufficient signal or to follow fast time-dependent chemical/physical phenomena (e.g., thermal transport). Temperature is measured radiometrically in the time domain using a streak camera or intensified CCD detector.

I will present the results of several types of experiments at synchrotron beamlines (GSECARS and EC P02.22 at DESY) and in our optical laboratory at GL CIW. These are measurements of thermal expansion, melting, thermal conductivity, and optical properties (including mid infrared), and also synthesis of new material in extreme P-T conditions.

I acknowledge the support of NSF, DARPA, Army Research Office, and Deep Carbon Observatory.

4.7. Fast temperature readout spectrometer for atomic dynamics measurement

Dongzhou Zhou

California Institute of Technology & Argonne National Laboratory

To capture the sample's transient temperature fluctuations and reduce uncertainties in melting temperatures, we have developed a Fast Temperature Readout (FasTeR) spectrometer in-line with nuclear resonant scattering measurements under extreme conditions at Sector 3-ID-B of the Advanced Photon Source at Argonne National Laboratory. Dedicated to determining the sample's temperature near its melting point, FasTeR features a fast readout rate (up to 400 Hz), high sensitivity, large dynamic range and well-constrained focus. This system replaces an intrinsically slow charge-coupled device (CCD) spectrometer with fast photomultiplier tubes and an optical dichroic filter array. FasTeR is capable of reading out temperatures about 1 to 2 magnitudes faster than the conventional CCD spectrometer, without sacrificing accuracy, and is especially suitable for dynamic measurements at extreme conditions. Working in parallel with the laser-heated diamond anvil cell, nuclear resonance scattering and X-ray diffraction, we have successfully used this system to measure the melting point temperature of ^{57}Fe and $^{57}Fe_{0.9}Ni_{0.1}$ at high pressure.

4.8. Time-resolved X-ray diffraction at APS Beamline 7ID

Donald A. Walko

Advanced Photon Source, Argonne National Laboratory

Beamline 7ID of the Advanced Photon Source specializes in time-resolved x-ray research, with a time resolution, given by the x-ray pulse duration, of about 100 ps. A variety of pump lasers allow flexibility in excitation wavelength and repetition rate for x-ray diffraction and spectroscopy experiments. Laser pulses can be synchronized to the fill pattern of the storage ring with electronically adjustable delays. We will give examples of time-resolved diffraction as applied to experiments in thermal transport, strain propagation, and photoinduced phase transitions. We will also discuss the issues involved in expanding time-resolved diffraction to samples in high-pressure environments.

5.1. Laue capabilities at HPCAT - towards time-resolved studies

D. Popov, C. Park, G. Shen

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White beam diffraction is potentially a powerful tool to investigate changes of microstructure due to increase of pressure. Diffraction data can be collected one-two orders faster if compare to using of monochromatic beam because multiple reflections can be recorded with an area detector simultaneously without need to rotate the sample. This in turn makes possible getting of both spatially and time resolved data during the same experiment with much better time resolution. Despite Laue diffraction is widely implemented in materials science and protein crystallography for high pressure area this is rather a new approach. Its possible applications include studies of mechanisms defining kinetics of pressure induces structural phase transition and phase transformations like crystal growth, recrystallization or melting of single crystals.

Time resolved high pressure white beam diffraction is one of the development projects of HPCAT. Its current status and future plans will be considered during the presentation. Experimental setup currently available at bending magnet based HPCAT 16BMB beamline allows doing real time Laue measurements on single-crystals in diamond anvil cells (DAC) with the best available time resolution in a range of seconds. Some software to index diffraction spots and calculate relative orientations of single-crystals using this setup was developed. Examples including observations of microstructural changes during pressure induced phase transitions will be shown in order to demonstrate currently available experimental and data reduction capabilities. In general such measurements are real time measurements and require fast data collection because pressure control during phase transitions is very challenging due to DAC instability and volume change of the sample.

5.2. Dynamic Laue diffraction: a probe for understanding the kinetics of strength and phase transitions

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Time-resolved polychromatic (Laue) diffraction using the brilliant, focused beams available at 3rd generation synchrotron sources comprises an unprecedented tool for the study of lattice relaxation and phase transitions in crystalline specimens. In this presentation, the fundamental platform is presented in the context of the APS. Several specific dynamic measurement platforms are described, including the kinetics of deviatoric stress relaxation, solid-solid phase transitions and twinning, and amorphousization/melting. Some of the unique challenges and areas in need of development to make the technique available to HP-CAT users are discussed. In all – and with the proper ancillary drivers (lasers, dynamic DAC, etc...) – this capability promises to advance our understanding of strength, equation of state, and dynamic phase diagrams for a wide array of materials.

5.3. 100 ps time-resolved white beam diffraction at BioCARS

Keith Moffat

Department of Biochemistry & Molecular Biology, Institute for Biophysical Dynamics and Center for Advanced Radiation Sources (CARS)
The University of Chicago

If a stationary crystal is illuminated by a polychromatic X-ray beam from an undulator or bending magnet source, Laue diffraction occurs (Moffat, 2007). By integration over X-ray wavelength, each diffracted beam automatically generates the integrated intensity necessary for subsequent structural analysis. This contrasts with the more common monochromatic diffraction methods in which the sample is rotated during the exposure to achieve integration over angle. The wide wavelength/energy range in Laue diffraction means that a substantial number of Bragg spots (reciprocal lattice points), lying between the Ewald spheres corresponding to Emin and Emax, diffract both simultaneously and strongly, since they satisfy Bragg's Law throughout the exposure. That is, Laue exposures are short.

At BioCARS sector 14 (Graber et al., 2011), either white or pink or monochromatic, mirror-focused radiation can be delivered to the sample. The beamline has a fast shutter train which can isolate either a single 100 ps X-ray pulse when APS is operated in the standard 24-bunch mode or in hybrid mode, or a train of pulses of any desired length. The shutter is phase-locked to the accelerator ring clock. Visible laser pulses can also be delivered, to enable pump-probe experiments in which a laser pulse (the pump) initiates a light-dependent reaction and after a controlled time delay an X-ray pulse (the probe) interrogates the structural state of the sample. Variation of the time delay enables the entire time course of the reaction to be traversed. The jitter in the pump-probe timing is <10ps, and the time resolution of overall experiments is <~100ps.

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5.4. Time-resolved X-ray emission spectroscopy and related techniques

Tsu-Chien Weng

SLAC National Accelerator Laboratory

The light-driven processes in 3d transition metal based photo-catalysts and photo-sensitizers is the key to control the photo-energy conversion. The detailed understanding of such non-equilibrium excited-state dynamics and their interplay with structural change is challenging. Here we present a femto-second resolution study on the spin dynamics in archetypal polypyridyl iron complexes upon photo-excitation. The excited-state charge and spin dynamics involved in spin crossover (the transition from a low- to a high-spin state) have long been a source of interest and controversy. XES measurements of iron complexes provides a clear means of characterizing the charge and spin on the metal centre, making it an ideal probe of ultrafast electron dynamics in coordination chemistry. Our recent optical pump x-ray probe study of trifluorene will also be present. This oligomer serves as a model to its polymeric counterpart polyfluorene for the investigating the exciton relaxation process. Some practical experimental issues will be discussed. We anticipate that the x-ray emission spectroscopy is a valuable tool for mapping in details the fundamental electronic excited-state dynamics that underpin many useful light-triggered molecular phenomena.

5.5. Fast detectors for time resolved studies – current status and new developments

Robert Bradford

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X-ray detectors continue to evolve rapidly, and new developments in the field will revolutionize detectors for time resolved high pressure science. In particular, new second generation hybrid pixel detectors will offer kilo-hertz frame rates and wide dynamic range. Progress in hi-z sensors (cadmium telluride, germanium, and gallium arsenide) will also greatly improve detectors for high energy x-rays. This talk will summarize developments from both commercial vendors and laboratory detector groups that are applicable for HPTR studies.

5.6. Laser-based dynamic compression of solids

Tom Duffy

Princeton University

Laser-based dynamic compression provides new opportunities to achieve ultrahigh pressure conditions in the laboratory. In this technique, high-powered laser beams are used to ablate a sample surface and by reaction a compression wave propagates through the material under study. By controlling the shape and duration of the laser pulse, either shock or ramp (shockless) compression can be produced. Molybdenum (Mo) is a technologically important transition metal that is used as a standard in static and dynamic compression experiments. However, significant unanswered questions and unresolved discrepancies remain about the high pressure-temperature phase diagram of this fundamental material. We have carried our laser-compression experiments on Mo to as high as 1000 GPa using x-ray diffraction as a diagnostic. Our results provide the first direct experimental determination of the crystal structure of Mo at these extreme conditions. We find that the body centered cubic (BCC) structure remains stable until shock melting occurs at about 400 GPa and under ramp loading the BCC structure is stable until 1000 GPa. Our results enable us to constrain the phase stability, melting curve, and equation of state of Mo to unprecedented levels of compression.

Our dynamic compression studies also have applications towards understanding the interior structures of extrasolar planets. Magnesium oxide (MgO) is likely to be a major constituent in the mantle of super-Earth planets. Ramp compression has been used to study MgO to 900 GPa and we have obtained the first direct evidence from x-ray diffraction for the rocksalt to cesium chloride phase transition near 600 GPa. In other experiments, we have measured the equation of state of diamond to record-breaking pressures up to 5000 GPa. These experiments have achieved pressures of Jupiter's core in the laboratory for the first time, and have implications for the interior structure of large planets, both within and outside our solar system.

5.7. Ultrafast x-ray scattering measurements from dynamically compressed solids

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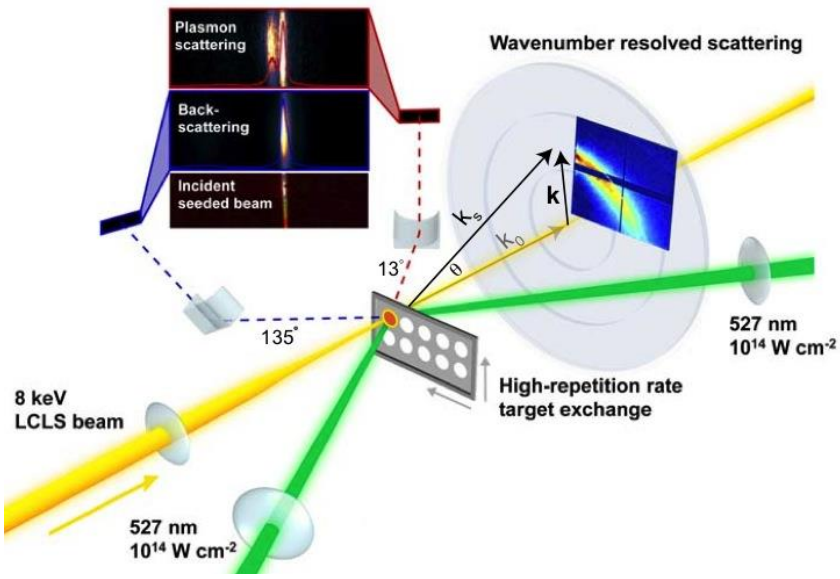
With the advent of the Matter in Extreme Conditions instrument at the Linac Coherent Light Source a world-unique experimental capability has become available to study the physics of dynamically compressed solids. Our new high-energy-density science program at SLAC is aimed to take advantage of x-ray pulses with the highest peak brightness available today. In a single shot, the x-ray beam delivers 10^{12} x-ray photons in 50 fs focused to a spot of order 1 µm. This capability allows us to measure plasmons in shock-compressed or ramp-compressed matter using nanosecond laser beams. For example, high-density aluminum has been compressed up to a mass density of 7 g/cm^3 with a free electron density of n_e = 4.7 x 1023cm^{-3} and a temperature of 20,000K. In these conditions, we visualize the density and pressure evolution across the melt line by resolving correlations up to distances comparable to the atomic size of aluminum. Our data allow direct determination of pressure for validating theoretical models for the thermodynamics at high pressure. We will show how LCLS data test our theoretical models of compressed matter and will discuss future plans for the study of hot and dense matter.



The figure shows a schematic of experimental setup at the Matter in Extreme Conditions (MEC) instrument at LCLS. The forward x-ray scattering spectrum shows inelastic scattering on plasmons. A CSPAD area detector observes the total wavenumber-resolved x-ray scattered intensity observing an intense feature due to ion-ion correlations.

5.8. A first laser shock experiment at the ESRF to probe Warm Dense Iron

F. Occelli[1], A. Sollier[1], E. Lescoute[1], L. Videau[1], P. Loubeyre[1],R. Torchio[2], O. Mathon[2], G. Berruyer[2], S. Pasternak[2], F. Perrin[2], S. Pascarelli[2], J. Headspith[3], W. Helsby[3], D. Eakins[4], S. Bland[4], D. Chapman[4], K. Mecseki[4], S. Rose[4], T. Vinci[5], M. Harmand[5] and A. Benuzzi[5]

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We report here the first laser shocked Fe K-edge dispersive EXAFS measurements on one of the ESRF's recently upgraded beamline (ID24) using a single 100ps x-ray pulse (in 4-bunches mode). Generally such experiments are carried out on a much bigger scale using high power kJ lasers where two orders of magnitude more energy are needed [1], but where the spotsize on target is in the mm range. Taking advantage of the x-ray focusing capabilities of the beamline (x-ray spot size in the 10µm range), we could focus our laser to a spotsize in the 100µm range on target permitting our portable 40J laser to provide a fluence (power/surface unit) of the same order of magnitude as what provide the large dedicated facilities permitting to reach P-T states in the same range.

Solid-solid and solid-liquid phase transitions in iron under extreme pressures and temperatures could therefore be observed using single shot XANES and EXAFS over spectral windows that were first 150 and then 300eV in width (depending on the beamline settings). The shock lifetime in the iron target was confined for a few nanoseconds using a pair of diamond windows, providing a time window sufficiently large for the 100ps synchrotron pulse to probe thermodynamically stable states reaching 370 GPa and 10000 K. The evolution of the thermodynamical conditions during the shock could be probed by varying the laser/x-ray pulse delay.

Being able to study hot, dense matter in this way offers valuable information on the electronic and local structure for the theoretical modelling of "exotic" states like the Warm Dense Matter (WDM) where most of the approximations used in condensed matter physics or in plasma physics break down [2]. The quality of the data collected on shocked Fe using a single X-ray pulse is similar

to that obtainable at ambient conditions. This first experiment demonstrates the feasibility of these studies at a synchrotron beamline, and opens many exciting opportunities for probing the local and electronic structure in very dense states of matter. Dynamic compression experiments thus will in the future become more accessible and profit from the extremely stable X-ray diagnostics of the synchrotron beamlines.

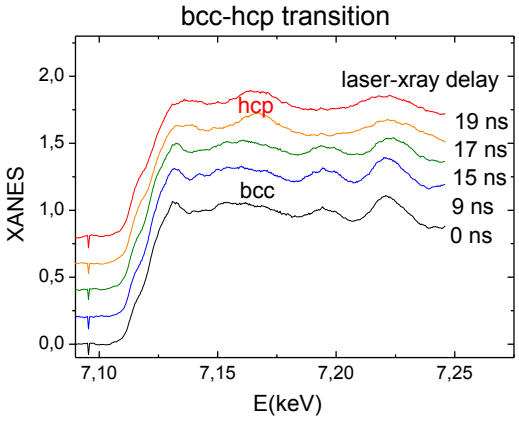

Figure 1. Laser-driven solid-solid phase transition in irons observed by X-ray absorption

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Techniques and Instrumentation Posters

HP1. High Pressure Collaborative Access Team at the Advanced Photon Source: an integrated facility for high pressure research

G. Boman, L. Bai, A. Bommannavar, P. Chow, R. Hrubiak, F. Humble, D. Ikuta, C. Kenney-Benson, Y. Kono, C. Lin, H. Mao, Y. Meng, C. Park, D. Popov, E. Rod, G. Shen, S. Sinogeikin, J. Smith, V. Struzhkin, Y. Xiao

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The High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source has been developed to optimize and integrate multiple novel synchrotron x-ray diffraction and x-ray spectroscopy probes, as well as complementary optical and electromagnetic probes, with diamond-anvil cell and Paris-Edinburg cell samples at high pressures and temperatures, and variable compression/decompression rates. By canting two undulators for the insertion device line, and branching the bending magnet line, HPCAT is able to operate simultaneously a total of four experimental stations in parallel with minimal compromise. This poster constitutes a visual overview of the beamline optics and station layouts at HPCAT. Established techniques are listed for experimental stations.

HP2. Structure and property measurements of liquids and amorphous solids in a Paris-Edinburgh cell at 16-BMB

Yoshio Kono[1], Curtis Kenney-Benson[1], Changyong Park[1], Guoyin Shen[1], and Yanbin Wang[2]

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Knowledge on the structure and physical properties of liquids at high pressures and high temperatures is important in understanding dynamics and evolution of the Earth and other planets. However, understanding the physics of liquid materials remains a challenge, especially under high pressure and high temperature conditions. We have recently developed an integrated setup for multi-angle energy dispersive x-ray diffraction, ultrasonic measurement, and falling sphere viscometer using a Paris-Edinburgh press at sector 16-BM-B, HPCAT, at the APS, for comprehensively studying structures and physical properties of liquids and amorphous materials at high pressures and high temperatures (Kono et al., 2014). The sector 16-BM-B is capable of amorphous and liquid structure measurements by using multi-angle energy dispersive x-ray diffraction technique at high pressure and high temperature conditions in a Paris-Edinburgh (PE) cell. The PE cell is capable of compressing large volume samples (typically >1 mm3) up to 7 GPa at temperatures exceed 2000 °C. In addition to the liquid and amorphous structure measurement capability, we have developed ultrasonic elastic wave velocity and falling sphere viscosity techniques in the PE cell. The elastic wave velocity is measured by ultrasonic pulse echo overlap method in conjunction with white x-ray radiography measurement for determining the wave travel distance (Kono et al., 2012). The falling sphere viscosity measurements are made with high-speed white x-ray radiography (>1000 frame/second) (Kono et al., 2013). The integration of liquid structure measurement with elastic wave velocity measurement and viscosity measurement in the PE cell provides a unique opportunity to investigate in situ correlation between microscopic structure and macroscopic properties of liquids and amorphous solids. Knowledge of the correlation will provide valuable constraints for modelling liquid properties at high pressures and high temperatures, and thus improving our understanding the nature of magmas at depths and the dynamics of the Earth's interior.

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Kono, Y., C. Park, C. Kenney-Benson, G. Shen, and Y. Wang, *Physics of the Earth and Planetary Interiors* **228**, 269-280 (2014).

HP3. Falling sphere viscosity measurements at high pressure in Paris-Edinburgh cell and diamond anvil cell

Yoshio Kono[1], Ross Hrubiak[1], Curtis Kenney-Benson[1], Changyong Park[1], Guoyin Shen[1], and Yanbin Wang[2]

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Viscosity is one of the most fundamental transport properties in liquid. Extensive studies have been made at ambient pressure using various techniques. However, the effect of pressure on the viscosity of liquids remains poorly understood partly due to the lack of experimental data. High-pressure falling-sphere viscosity measurement was first developed in an effort to measure viscosities of molten earth materials. These measurements were confined to highly viscous materials due to the limited imaging rate (typically 30-60 frames/second and up to 125 frames/second) in common x-ray radiography apparatus. As a consequence, it was difficult to investigate low viscosity liquids such as metals and salts whose viscosities are around 1 mPa s or less at ambient pressure. We recently have developed an advanced technique of falling sphere viscosity measurement using ultrafast X-ray imaging at high pressures, which enabled us to study viscosity of low viscous liquids at high pressures. The new technique has wide ranging applications not only in earth and planetary sciences but also in physics, chemistry, and material sciences, because viscosity of liquid is important not only in earth science areas (e.g., hydrology, volcanology, and study of earth and planetary interiors) but also in many industrial processes (e.g., metallurgical manufacturing, glass manufacturing, forming of polymers, and food production). In this poster, we will introduce falling sphere viscosity measurement not only in Paris-Edinburgh cell but also in diamond anvil cell. In addition, we will introduce a recent study of viscosity measurement for carbonate melt at high pressures (Kono et al., 2014).

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HP4. White beam diffraction setup at HPCAT 16-BMB beamline

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Experimental white beam diffraction setup currently available at HPCAT 16BMB beamline is intended for time resolved Laue measurements of microstructure in diamond anvil cells (DAC) in transmitted (when area detector is almost perpendicular to the beam) and 90° (when area detector is almost parallel to the beam) geometries. Intermediate detector positions between 0 and 90 degrees are also available. X-ray beam is focused down to 5-10μm^2 using KB-mirrors. Energy range 5-70kEv is much larger than at typical white beam diffraction dedicated facilities that provide reasonable access to reciprocal space despite of the limitations introduced by the DACs. Perkin Elmer area detector is typically used for data collection due to its short readout time of 133.2 ms. Si(111) channel cut monochromator can be moved in and out of the incident beam providing monochromatic beam at the sample position exchangeable with the white beam. This monochromatic beam is mainly implemented to measure d-values of reflections for reliable indexation of Laue diffraction patterns. As collection of diffraction data with the monochromatic beam requires much longer time than using white beam typically monochromatic data is collected before and after the studied microstructural changes. Based on known indices variations of crystal morphology, lattice rotations, texture and strain are analyzed by Laue diffraction. The monochromator is also used to calibrate sample to detector distance and detector tilt in transmitted geometry with CeO_2 powder standard. Some examples of real time microstructural measurements will be presented.

HP5. HPCAT 16-BMD: the micro-XRD and XANES beamline for high-pressure research

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The HPCAT 16BM-D beamline is dedicated to high-pressure research with single crystal X-ray diffraction (SXD), powder X-ray diffraction, and transmission mode X-ray Absorption Near Edge Structure (XANES) (6-45 keV) techniques. The setup is highly flexible to account for various experimental requirements including resistive heating and cryogenic cooling as well as ambient compression.

For micro-X-ray diffraction, the monochromatic incident beam is focused to 12-15 μm vertical and 4-5 μm horizontal (FWHM) by Kirkpatrick-Baez type mirrors. The typical angular resolution, limited by the beam divergence and the pixel resolution of the area detector, reaches approximately 2×10^{-3} in terms of the finest $\Delta q/q$ (i.e., the values at the highest q). The instrumental broadening can be adjusted through use of different detectors (e.g., MAR345 Image Plate detector, Perkin-Elmer a-Si array detector, Pilatus 1M pixel array detector, MAR CCD detector, etc.) and their distances so that the diffraction experiment can be optimized to the user's requirement.

Transmission mode XANES setup is readily combined with the optimized micro-XRD setup. For absorption spectroscopy, the full energy resolution, $\Delta E/E$, of the monochromatic beam ranges from 3 to 8×10^{-4} over a 6-45 keV energy range. The detector configuration can be quickly converted from a large area detector for XRD measurement to gas ion chambers for XANES measurements without losing beam alignment or altering the sample condition. The fixed-exit, pseudo channel-cut Si 111 monochromator and the achromatic beam focusing optics hold the beam position and size stable within the ~40 μm pinhole diameter. A mount holding both the monitor and detector ion-chambers is pneumatically translated in and out to ensure reproducible XANES measurement geometry. The switchable configuration streamlines combined XRD+XANES data collection by eliminating the need to realign the beamline optics or the sample position between measurements.

HP6. Angle dispersive diffraction capabilities at extreme conditions at 16-IDB

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16 ID-B, an undulator-based monochromatic XRD station, is a unique facility for micro-focused angle dispersive powder and single crystal X-ray diffraction under extreme PT conditions. 16-IDB is designed for performing high-pressure experiments at variety of temperature conditions – from double-sided laser heating (1000-s K) through resistive heating (100-s K) and down to cryogenic (4K) temperatures, and provides unique experimental capabilities for structural studies of materials under extreme conditions at megabar pressures.

Recently 16-IDB station has undergone a serious upgrade. First, now ID beamline hosts two undulators in canted mode, with one undulator serving the 16-IDB station independently of 16-IDD (spectroscopy beamline). Second, the water cooler Si220 Branching monochromator was replaced with a new 3-crystal (Si111, Si220, Si331) liquid nitrogen (LN) cooled monochromator with piezo-stabilization capability. The new monochromator expanded the energy range from ~25-35 keV to 12-43 keV. Also LN cooled monochromator provides the beam with much better divergence, which resulted in ~5 times increase in beam flux on the sample (up to $3x10^{11}$ p/s), and significantly improved beam focusing. Further 3x increase in flux comes from replacement of 200 mm KB mirror with a new KB mirror system with 320 mm mirrors. This upgrade in conjunction with newly acquired fast Pilatus 1M detector allows not only improvement of the efficiency and quality of diffraction measurements, but also allowed to effectively perform time resolved fast dynamic experiments such as diffraction combined with pulsed and power-modulated laser heating and extremely high strain rate unidirectional and cyclic compression-decompression experiments. The enhanced X-ray source also has impact on several areas of high-pressure research including high resolution XRD, low-Z material study at Mbar pressures and amorphous material research, to name a few. The overall description of the station, beam characteristics, detectors and some experimental setups are described in the poster.

HP7. New developments at 16-IDB using CW and pulse laser-heated DAC for studies under extreme conditions

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Laser-heated diamond anvil cell (DAC) combined with in-situ synchrotron x-ray radiation is a unique and powerful experiment technique for structural properties under static ultrahigh-PT conditions. In the past two decades, it has evolved into a reliable, effective and user-friendly experimental technique at synchrotron beamlines. However, majority applications are in the areas of phase transition, materials synthesis, and sample annealing for PV EOS measurement. High pressure melting study using synchrotron x-ray has been complicated by several issues including melt containment, lateral temperature gradient, and maintaining the exact alignment of melting spot and x-ray beam. P-V-T EOS study is another challenging area that requires the exact alignment of heating, x-ray and temperature measurement spots at all the time of the experiment, which is not always guaranteed in the conventional systems commonly used at synchrotron beamlines. To advance research in these challenging areas, we have been focusing on developing capabilities that address these specific issues. This presentation summarizes what have been established at 16IDB in recent years, including (1) in-situ heating spot variation (flat top area 4 to > 60 micron, FWHM 9 to > 120 microns) to provide effective and uniform heating on various-sized samples in diamond anvil cell; (2) mirror pinhole setup to allow experimenters directly viewing the image of spectrograph pinhole where the temperature sampling area is defined in temperature measurement, relative to the heating spot and x-ray beam position and to make adjustment if needed; and (3) synchronized short-pulse heating to reduce the exposure of the sample and cell to extreme conditions and to allow fast data capture and time-resolved studies under high PT conditions. These new capabilities have demonstrated clear benefit and providing new opportunities in areas including high-pressure melting, P-V-T EOS, chemical reaction and time resolved studies.

HP8. High pressure X-ray emission spectroscopy and nuclear resonant scattering at 16-IDD

Y.M. Xiao, P. Chow, G. Boman, L.G. Bai, E. Rod, C. Kenney-Benson, A. Bommannavar and G.Y. Shen

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As part of a third generation synchrotron radiation beamline dedicated to high pressure research, 16-IDD of HPCAT at the Advanced Photon Source engages in X-ray spectroscopy research of samples under high pressures, typically in diamond anvil cells (DAC) [1].

The spectroscopy line consists of IDA with a newly installed liquid nitrogen cooled Si (111) double crystal monochromator with ~1eV energy resolution, transport lines; IDC with an interchangeable high-resolution monochromator at ~2meV energy resolution and two 1-meter K-B mirrors; and IDD, the experiment station, with a 2.7 meter 0-90 degree horizontal spectrometer and a dedicated XES setup. Typical beam size at sample position is ~30(V) x 50 (H) μm^2 at FWHM when using meter-long KB mirrors, smaller beam size (3x5 μm^2) can be achieved by using a pair of 200mm KB mirrors.

Current techniques includes X-ray emission spectroscopy (also resonant X-ray emission spectroscopy), inelastic X-ray scattering (also X-ray Raman scattering) and nuclear resonant scattering (nuclear resonant inelastic X-ray scattering and nuclear forward scattering).

In this poster, we will mainly focus on X-ray emission Spectroscopy and Nuclear resonant scattering. Examples of high-pressure studies and recent developments will be discussed in details.

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HP9. X-ray Raman and the study of electronic excitations at 16-IDD

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When X-rays impinge on a sample, many processes can occur. In inelastic X-ray Spectroscopy, photons exchange energy and momentum (change direction) with electrons in the sample and scatter from the sample. The double differential cross-section, which is proportional to the number of photons with a given energy, within an energy range, scattered into a solid angle, is measured. The spectrometer is designed to detect scattered photons of energies of interest with an energy resolution and momentum transfer corresponding to the physical process of interest. So, if the photon energy-loss on the order of the binding energy of electrons of low-Z elements and energy resolution on the order of 1 eV, a measurement of the X-ray scattering cross-section is called X-ray Raman Scattering. Its spectrum is similar to that of Soft X-ray Absorption [1]. X-ray Raman yields information about the bonding of electrons and the electronic structure (energy levels and occupation of orbitals). The geometry of scattering can be selected to probe different bonding directions, for example in layered materials. If the photon energy-loss is on the order of a fraction of eV to tens of eV, one can study valance excitations and plasmons (collective excitations of the electron gas) with scattering close to the forward direction. This falls under the category of the spectroscopy of electronic excitations [2].

Presently, HPCAT has a spectrometer for the study of electronic excitations at low momentum transfer and X-ray Raman at larger scattering angles with about 1 eV energy resolution. We employ backscattering bent or diced Si(555) analyzers which scatter photons into a detector close to the sample. Multiple analyzers can increase the collection solid angle for low count-rate measurements. The incoming energy is scanned by the monochromator, with analyzer/detector geometry fixed to measure the Si(555) backscattering energy. The difference between the incoming energy selected by the monochromator and the fixed backscattering energy (9886 eV) is the energy loss of the photon.

HPCAT provides a facility for the study of electronic excitations at low momentum transfer and X-ray Raman at larger scattering angles with 1.4 eV energy resolution. Under development is the use of polycapillary optics to discriminate background due to gasket scattering and medium energy resolution energy loss scattering capability at 10 keV.

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HP10. Apparatus and techniques for time-resolved synchrotron x-ray diffraction using diamond anvil cells

Jesse S. Smith, Chuanlong Lin, Ligang Bai, Eric Rod, Stanislav Sinogeikin, and Guoyin Shen

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Complementary advances in synchrotron sources, x-ray optics, area detectors, and sample environment control have recently made possible many time-resolved experimental techniques for studying materials at extreme pressure and temperature conditions. The High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source has made a sustained effort to assemble a powerful collection of high-pressure apparatus for time-resolved research, and considerable time has been invested in developing techniques for collecting high-quality time-resolved x-ray scattering data.

Techniques currently available or under development include (but are not limited to) pulsed/ramp laser heating for thermal equation of state and melting experiments; rapid, controlled sample compression/decompression—with rates ranging from less than a GPa/s to extreme compression in excess of a TPa/s—for synthesis/observation of metastable high pressure phases; high speed imaging for measuring viscosity at high pressure and temperature; and Laue (white beam) measurements meant to study phase transitions as they unfold. Herein we describe the equipment, measurement process, and early results from some of these techniques.

HP11. Fast compression/decompression: an overview of several experimental approaches with accompanying examples

Jesse Smith, Chuanlong Lin, Ligang Bai, Eric Rod, Stanislav Sinogeikin, and Guoyin Shen

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Fast compression/decompression constitutes one of the fundamental technical approaches to time-resolved high pressure research in the diamond anvil cell. Through careful choice of pressure generating apparatus and relevant time scale, a number of important scientific challenges can be address including, for example, non-equilibrium transformations and phase boundaries, unusual thermodynamic pathways to metastable phases, and system-dependent nucleation rates and crystal growth. In this work we show preliminary results from a number of different scientific studies, each taking advantage of a particular apparatus and compression rate to measure, for example, equations of state at ambient and elevated temperatures, high sample compression and strain rates in materials, and compression-rate dependent sample stress and subsequent relaxation.

HP12. Kinetics of phase transition in KCl and NaCl under dynamic (de)compression

Chuanlong Lin, Jesse Smith, Ligang Bai, Yoshio Kono, Curtis Kenney-Benson, Eric Rod, Stanislav Sinogeikin, and Guoyin Shen

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The kinetics of the B1-B2 phase transition in KCl and NaCl were studied using high-pressure time-resolved and dynamic compression-decompression techniques in Diamond Anvil Cell with dynamic pressure modulation. We found that the grain size, distribution and grain number are dependent on (de)compression rates in the process of nucleation and growth of the B1-B2 (or reverse B2-B1) transition in KCl. The pressure range of the hysteresis loop is found to be strongly dependent on compression-decompression rate, where faster rate shifts onset of the phase transition to higher/lower pressure resulting in over-(de)pressurization and broader hysteresis loop.

From the x-ray diffraction patterns, we obtain the time-dependent volume fraction of B1 and B2 phases. Combined with transformation rates, we can study the competition between activation energy and driving force in the phase transition process of KCl and NaCl.

HP13. Study of phase transition pathways, metastable phases, melting and crystallization using time-resolved x-ray diffraction and dynamic (de)compression techniques

Chuanlong Lin, Jesse Smith, Eric Rod, Stanislav Sinogeikin, Guoyin Shen

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High-pressure Diamond Anvil Cell – based time-resolved x-ray diffraction and dynamic (de)compression techniques at High Pressure Collaborative Access Team (HPCAT) provide the possibility to observe (de)compression-rate dependence of phase transition pathways, pressure-quenching of metastable high-pressure phases, as well as kinetics of phase transformation, melting and crystallization. Here we present some examples of our recent experimental results of dynamic (de)compression in common elements and compounds – silicon, gallium, and water.

For water, we observed compression-rate dependence of phase transition pathways directly using time-resolved x-ray diffraction. We found that at large compression rates water transformed to ice VII directly without going through ice VI phase, while at slower compression rates it crystallizes to ice VI phase first and only then to ice VII.

It is well known that silicon has several metastable phases at low pressures. We consistently observe different metastable phases (crystalline and amorphous) or mixture of them at different controlled de-compression rates, and these metastable phases can be kept for a long time at ambient conditions.

In pure metal Gallium we studied the process of cyclic melting and crystallization under cyclic compression - decompression. We found that the compression rate has a remarkable effect on the phase transition / crystallization pressure (over-pressurization), while during decompression the rate has a negligible effect on transition / melting pressure (over-depressurization).

In summary, the combination of time-resolved XRD and dynamic (de)compression techniques is a powerful tool for studying the rate dependence of phase transition pathways, metastable phases, and process of melting and crystallization in the high-pressure field. These techniques are currently available at HPCAT.

HP14. Supporting facilities at HPCAT: Optical systems

Stanislav Sinogeikin, Curtis Kenney-Benson, Ross Hrubiak, Jesse Smith, Yue Meng, Eric Rod, Guoyin Shen

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High Pressure Collaborative Access Team (HPCAT) is dedicated to advancing cutting-edge, multidisciplinary, high-pressure science and technology using synchrotron radiation. At HPCAT an array of novel x-ray diffraction and spectroscopic techniques has been integrated with high pressure and extreme temperature instrumentation. Over the last several years a number of supporting facilities have been developed and implemented to expand the available P-T range of the experimental conditions, increase efficiency and productivity of the beamlines, improve the quality of experimental data, and integrate additional methods of sample characterization with synchrotron investigations.

A considerable effort was put into developing instrumentation, which allows remote and automatic pressure control in diamond anvil cells (DACs) during synchrotron experiments. Such devises require fast on-line pressure measurement capabilities. We have designed a number of portable online optical systems for ruby fluorescence pressure measurements and in-situ Raman sample characterization during synchrotron experiments. Now online optical systems, capable of measuring pressure in cryostats, resistively heated cells, and at room temperature, are available at all four experimental stations at HPCAT. Combined with remote pressure control systems, they significantly increase productivity of beamlines during high-pressure experiments.

We have also designed and implemented a number of offline optical systems for pressure and Raman measurements, offline laser heating, IR laser micro-drilling/micromachining system for sample/gasket preparation (see Poster HP15), and so on. These systems are operational and are intensively used by the users of HPCAT and other APS beamlines. Some of these systems have really unique capabilities. For example the offline ruby system is now capable of collecting and recording ruby spectra at 200 Hz frequency (5 ms interval). This allows accurate characterization of decompression rate in fast pressure jump / pressure quench offline experiments. The newly redesigned offline double-sided laser heating system with variable focus and accurate temperature measurement capability allows stress annealing and high pressure-high temperature sample synthesis in Diamond Anvil Cell, and at the same time serves as a testing ground for new setups and instrumentation in traditional and pulsed / power modulated laser heating for melting and equation of state studies.

HP15. Laser drilling and micromachining system at HPCAT

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We have designed and constructed a new system for micromachining parts and sample assemblies for diamond anvil cells and general user operations at the Advanced Photon Source. The new drill system uses a pulsed laser of 400 picosecond pulse duration, ablating materials without thermal melting, thus leaving a clear edge after drilling. With optics designed for a tight focus, the system can drill any size holes larger than 3 micrometers in diameter. Unlike a standard EDM drill, the new laser drill system allows drilling non-conductive materials such as amorphous boron and silicon carbide gaskets, diamond, oxides and other materials including organic materials (e.g. kapton). In addition, an important advantage of the new system is that it includes an air-tight sample enclosure which allows the laser machining to be done in an inert gas atmosphere to prevent oxidation of air sensitive materials. The air-tight sample enclosure is also useful for drilling materials with known health risks (e.g. beryllium). In-house remote computer control software with a graphical interface enables to drilling/machining of custom 2D and 3D shapes. The video alignment system of the laser drill includes the capability to view the samples with two distinct zoom settings, i.e. low and magnification. Dual, low and high, magnification settings help speed up the typical user operation of locating the region of interest on the samples and accurate alignment of the samples. The laser drill system was designed in a Class 1 laser enclosure, i.e. it includes laser safety interlocks and computer controls and allows for routine visiting user operation at the Advanced Photon Source (APS). We present several examples of typical applications for the new laser drill system.

HP16. Online pressure control at HPCAT

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The ability to change the pressure in a diamond anvil cell (DAC) remotely is a key factor in increasing productivity in high pressure x-ray measurements of all types. HPCAT has developed mechanical gearboxes, gas diaphragm systems, and piezo drives for pressure control under experimental conditions ranging from cryogenic to laser heated temperatures. These systems allow for static and dynamic (controlled ramp, pressure jump, cyclic pressure modulation) remote pressure control without the need to access the experimental hutch. The capabilities of each of these systems are outlined and examples of their use with various DACs are illustrated.

HP17. Online temperature control at HPCAT

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In-situ temperature control is a critical component of many experiments conducted at the Advanced Photon Source. HPCAT develops and maintains a selection of flexible cryostats and resistive heaters which can be configured to work with a wide variety of diamond anvil cells (DACs). These temperature control devices can be used with variety of x-ray techniques – single crystal and powder x-ray diffraction, spectroscopy and inelastic techniques as well as fast imaging. The cryostats allow temperatures down to 4 K, while with resistive heating the samples under pressure can be heated to at least 1000K. The current capabilities of cryostat and resistive heaters are outlined in the poster and examples of their use with various DACs are illustrated.

Contributed Posters

CP1. Effect of external pressure on the octahedral distortions in multiferroic *R*CrO_3 (R = rare-earth)

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Rare earth chromites *R*CrO_3 (*R* = Rare-earth) are interesting class of perovskite oxides with an intriguing multiferroic property which is rooted from 4f-3d magnetic interactions [1,2] which can be altered either by doping (chemical pressure) or by external pressure. We have aimed at studying the structural aspects of these materials as a function of pressure in order to predict room temperature multiferroic *R*CrO_3.

We have studied the effect of external pressure on the octahedral distortions in rare-earth chromites (*R*CrO_3; R= Lu, Tb, Gd, Eu, Sm) using Raman scattering and synchrotron x-ray powder diffraction up to 20 GPa [3]. Our studies reveal that the octahedral distortions in *R*CrO_3 increase with pressure at a rate which decreases with increase in R-ion size from *Lu* to *Sm*. The root cause for this effect is found to be the reduction in the compression of *R*O_{12} polyhedra with a corresponding increase in the R-ion radii. From the Raman studies we predicted a critical R-ion radius above which we expect the octahedral distortions in *R*CrO_3 reduce with increase in pressure leading to the symmetry lowering phase transition as seen in $LaCrO_3$. These results were further supported by the pressure dependent structural studies on *R*CrO_3 (R=Gd, Eu, Sm) [3]. Also, our results suggest that the pressure dependence of Néel temperature of Chromium (T_N^{Cr}) in *R*CrO_3 is mostly affected by the compression of Cr-O bonds rather than the alteration of octahedral tilts.

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CP2. Development of a New Experimental Capability at 3-ID of the APS: Nuclear Resonant Inelastic X-ray Scattering at High Pressure and Low Temperature

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We present the development of a new capability of nuclear resonant inelastic x-ray scattering under high pressure using diamond anvil cell (DAC) and at low temperature at 3-ID of the Advanced Photon Source, Argonne National Laboratory. The new technique can be applied to studies of phonon contribution to pressure- and temperature-induced spin, magnetic, superconducting, and metal-insulator transitions in resonant isotope-bearing materials, such as magnetic transitions in complex Fe-bearing systems to clarify different Fe site assignments and recently discovered Fe-pnictides and Fe-chalcogenides in order understand the contribution of spin, magnetism, valence and phonons to superconductivity.

CP3. Constraints on the Size of Io's Core

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Io is the innermost moon of Jupiter and the most geologically active body in our solar system. The exploration of this Jovian satellite was revolutionized by the Galileo mission which provided accurate measurements of the mean density and the moment of inertia factor. These results showed that Io is a differentiated body with a silicate mantle and a dense core. Assuming an Fe-FeS core composition, previous models for Io's interior structure have been constructed but these models use only estimated values for the densities of Fe and Fe-FeS compositions [1] under Io interior conditions. In this study, we reevaluate Io interior models using new experimental data for core components from recent high pressure-temperature experimental studies [2-7]. Recent P-V-T equation of state studies [2] of γ-Fe indicate that the density of pure iron is significantly lower than used in previous Io models [1, 8]. For Fe-FeS compositions, existing studies (3-7) disagree stronglyregarding the high-pressure densities. We evaluate both high- and low-density models for Io's interior. We find that a pure iron core would extend to 38% of Io's radius (compared with 33% in earlier studies [1, 8]. For the Fe-FeS eutectic core the radius may range from 47% to 58% of the planet's radius depending on which set of experimental data is used. These compare with earlier studies [8], which estimated a core radius of 50% for the Fe-FeS composition. These results highlight the importance of further high P-T experimental studies in the Fe-FeS system to better constrain Io interior models.

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CP4. High pressure phase transition in (Mg,Mn)O

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Manganese oxide is a minor component of the Earth's lower mantle, and partitioning of manganese between metal and silicates/oxides at high pressures and temperatures has been used to constrain the conditions of Earth's core formation. Therefore, it is important to understand the physical and chemical properties of Mn in (Mg,Mn)O at the high pressure conditions of Earth's mantle. Endmember MnO undergoes a spin transition at high pressures. The associated volume change in the MnO component of an oxide will affect the metal-oxide partitioning of Mn, possibly limiting our ability to extrapolate experimental partitioning data to higher pressures. We have studied a manganese–magnesium oxide containing 20 mol% Mn using both synchrotron X-ray diffraction and visible absorption spectroscopy in a diamond anvil cell, at beamline 12.2.2 of the Advanced Light Source and beamline U2A of the National Synchrotron Light Source. Pressure was measured using gold X-ray diffraction, ruby fluorescence, and diamond Raman spectroscopy, and neon was used as the pressure medium to generate quasi-hydrostatic conditions. Our X-ray data extend to 140 GPa at room temperature, with a change in compressibility occurring at 55 GPa. The sample changes color and becomes opaque beyond this pressure, which we have documented in photomicrographs and quantified using optical absorption measurements to 85 GPa. Thermodynamic modeling based on our results allows for calculations of phase diagrams in the MnO–MgO system, which indicate that (Mg,Mn)O in the Earth's lower mantle is in the low spin state.

CP5. New high-pressure orthorhombic Imma Ca-Fe silicate perovskite with hedenbergite composition

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Pyroxenes are important minerals in both mantle and subduction zones by constituting up to 20% of the upper mantle and up to 80% of basalt. At the pressure and temperature conditions of upper mantle, pyroxenes gradually dissolve into garnet, and then break down into $(Mg,Fe)SiO_3$ and $CaSiO_3$ perovskites. At temperatures well below the geotherm, however, pyroxenes can be metastably compressed to pressures as high as 50 GPa, thus making possible their preservation in the subducted slab in the transition zone, and perhaps even into the lower mantle. Several new metastable high-pressure phases of pyroxene have been discovered recently. For example diopside was found to transform to a high-pressure form with six-coordinated silicon at 50 GPa and room temperature [1], while enstatite undergoes a symmetry change to a P21/c orthopyroxene form at ~12 GPa [2], followed by further transformations affecting the tetrahedral chain kinking [3]. Here, with single-crystal x-ray diffraction, we report a discovery of a new metastable phase transition of Ca, Fe-rich clinopyroxene ($Ca_{1.00}(Fe^{2+}_{0.67}Mg_{0.17}Mn^{2+}_{0.16})_{\Sigma=1}Si_{2.00}O_6$) at ~35 GPa and ambient temperature to a previously-unknown orthorhombic Imma perovskite phase, accompanied by ~10% density increase. This new transformation is consistent with earlier observation of discontinuous change in the Fe coordination environment obtained from high-pressure Moesbauer spectroscopy experiments [4].

$(Mg,Fe)SiO_3$ and $CaSiO_3$ perovskites have different crystal symmetries and are generally expected to be immiscible in the lower mantle, however, a number of studies (e.x. [5][6][7]) suggest possibilities of incorporation of Fe into $CaSiO_3$ through various structural mechanisms. Proper understanding of these processes is obscured by the fact that $CaSiO_3$-pv is not quenchable, and its exact structure at higher pressure is not known (it is expected to undergo a distortion from the ideal cubic symmetry). The transformation found in our experiments proceeds though a single-crystal to single-crystal mechanism, and the structure of the Imma phase has been solved by high-pressure single-crystal diffraction, thus offering an unprecedented opportunity to study fine structural details of the new phase. Our results support the notion of possibility of incorporation of

iron into the Ca-pervoskite in the subduction slab at lower mantle conditions and suggest that the structure and symmetry as well as physical properties of $CaSiO_3$-pv may be affected by this process.

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CP6. Alpha-omega structural stability in Zirconium as a function of compression rate

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Investigation of pressure-temperature (P-T) phase diagram has been focus of research for over a century. High P-T research is broadly categorized into two categories: static (e.g. diamond anvil cell (DAC), large volume press, etc.) and dynamic (e.g. gas guns, laser drive compression, etc.) pressure techniques. The first of these involves nearly steady state conditions, where the stress state (pressure) is changed slowly and held over long periods of time, while second involves rapid changes that are often accompanied by large changes in temperature. Although such data gives a good insight into properties of materials, it largely leaves the kinetics associated with any high pressure polymorphs out of the equation, as they are too fast to be seen in traditional DAC's and too slow to be seen in shock studies. To this end, we have used several more recently developed techniques, such as pulsed pressure [1] and dynamic DAC's [2], to study intermediate strain rates on zirconium (Zr). This work will present some of our findings with regard to the alpha to omega structural transition in Zr and explore implications that variable strain/loading rates can have on material properties.

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CP7. Unraveling the structural complexity of high-pressure orthorhombic iron oxides

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Iron oxides have a major petrological, economical and technological importance. Their role as oxygen buffers, in differentiation processes and as magnetic phases summarize the iron oxides critical importance in most petrological contests, independently of their abundance.

The recent discovery of a new compound in the Fe-O system, Fe_4O_5 [1], reshaped our assumptions on the behavior of iron oxides in the Earth's deep interior, where phases of FeO and Fe_3O_4 were considered the sole plausible players. Fe_4O_5 is stable at pressures greater than about 10 GPa and is a plausible accessory mineral of the deep Earth. Other studies found that Fe_4O_5 may be stable in a wide stoichiometry range [2] and can accept a wide extent of isomorphic substitutions [3]. Finding a new compound in one of the most investigated binary systems and in a pressure and temperature range that is experimentally accessible for decades is surprising. The discovery of Fe_4O_5 relies on high resolution high pressure microdiffraction mapping exploiting the brilliant APS x-ray source and the in-situ laser heating equipment available at HPCAT and GSECARS. We will present the procedures that led to the discovery of Fe_4O_5 reported in a recent procedure video-publication [4]. Rastering the samples with still diffraction patterns provide information on the sample phase variability and is used to identify the best locations for the crystallographic analysis. We combined powder, single crystal and multigrain diffraction strategies within samples that are yet only about 100 µm large and less than 10 µm thick. This strategy can provide a robust identification and characterization of phases synthesized at high P-T that might be relatively complex.

We will present some of the latest results revealing an Fe-O system that shows at high pressure an un-expected complexity including important degrees of disorder, as evidenced by diffuse scattering.

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CP8. Viscosity measurements of eclogite melt up to 5.6 GPa and 2000 K

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The melting and rheology of eclogite is critical for understanding the dynamics of the subduction zone and plate tectonic. Viscosity of eclogite melt as a function of depth is a key parameter to model the long-term chemical evolution of the subduction zone and volcanic magma. It also plays an important role in earthquakes and continent formation. However, the viscosity and rheology of eclogite melt remain poorly understood at high pressure and temperature. In this study, we have carried out in-situ falling-sphere viscometry measurements to determine the viscosity of eclogite melt (with 47.7wt% SiO_2 and ~0.5%H_2O) from 1.3 to 5.6 GPa at temperatures between 1500 and 2000 K, using the Paris-Edinburgh cell at the 16-BM-B beamline of the High Pressure Collaborative Access Team (HPCAT) at the Advanced Photon Source. The measured viscosity ranges from 1.43 Pa S to 0.13 Pa S at the pressure of 1.3 to 5.6 GPa and temperature of 1500K to 2000K. At 1.3 GPa and 1500K, the viscosity of eclogite melt is 1.43 Pa S. The viscosity of eclogite melt decreases between 1.3 and 5.6 GPa at 2000K. At 1.4 GPa and 2000K the viscosity is 0.43 Pa·s, whereas at 5.6 GPa and 2000 K it is 0.13Pa·s. No minimum viscosity of eclogite was found in the measured pressure range up to 5.6 GPa.

CP9. Elasticity of the highly-textured hcp-Fe at high pressures

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Down to the Earth's core, iron (Fe) is the most abundant component. Knowledge of the elastic properties of Fe is thus essential for understanding the velocity profiles of the Earth's core with respect to seismic observations. The elastic anisotropy of inner core, exhibiting Vp anisotropy of 3-4%, is mostly believed to be a result of the lattice preferred orientations of the elastically anisotropic iron alloy crystals [1]. However, the anisotropy of the iron crystals at relevant conditions of the Earth's core has remained mostly theoretical [2,3], and high-pressure experimental data remain scarce [4]. Discrepancy in the magnitude and shape of the elastic anisotropy among various theoretical calculations and high-pressure experiments also remains to be resolved, demonstrating the need for further investigations. With the advent of the high-energy resolution inelastic X-ray scattering (HERIX) at XOR3, APS employing higher-energy resolution and smaller beamsize, small uncertainty (~1%) in Vp has been recently achieved [5]. Such data quality is critical to understand the Vp anisotropy of the inner core. The HERIX measurements on the apparent Vp anisotropy of textured hcp-Fe were carried out as a function of orientations at high pressure up to 160 GPa in a diamond anvil cell. Combining with the Monte-Carlo simulation, we try to determine the single-crystal elastic constants of hcp-Fe at core pressures, which are experimentally unavailable due to the lack of hcp-Fe single crystals. These novel results would provide first hand, reliable constraints on the elastic properties of Fe at relevant pressures and the seismic anisotropy of the inner core.

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CP10. The development of high-pressure techniques for time-resolved experiments at BSRF

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Some techniques for time-resolved high-pressure experiments using synchrotron radiation are being developed at BSRF. A loading mechanism with electromechanical piezoelectric actuators has been designed, which can rapidly control the sample compression/decompression and match to conventional symmetric DACs. The preliminary test demonstrates the compression rate up to 2600 GPa per second. Using this device and Pilatus detector we have obtained the X-ray diffraction data of PbS under different compression rate, the first time-resolved high pressure experiment at BSRF.

As an earlier result at BSRF, we have combined a dynamic DAC with EXAFS method to perform the DiffEXAFS experiments under the modulation of high pressure. The results on ZnSe at initial pressure of 4.8 GPa revealed sensitivity to atomic displacements of one order higher in magnitude than that of conventional EXAFS.

We are also making preparations for the setting up of a pulse laser heating system at 4W2 beamline of BSRF. This will enable the measurements of thermal equation of state. Preliminary design of the system will be introduced.

CP11. Rapid high-pressure high-temperature phase diagram of Gadolinium using a boron-doped heater anvil

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Boron-doped designer heater anvils have now been used to collect spectra for an entire range of pressures and temperatures in Gadolinium (up to 7 GPa and 600K) on a quasi-hydrostatically loaded sample. This technique allows for the temperature of the diamond itself to be precisely controlled and rapidly changed by the simple application of an externally-controlled direct current. The collected data set covers the entire range in P-T space with 0.2 GPa and 10K resolutions. Additionally, the temperature cycling allows us to investigate differences in material behavior upon different heating powers, to compare behavior on heating and cooling, and to observe differences between compression and decompression.

CP12. Properties of alkali-doped aromatic hydrocarbons under extreme pressures

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While structures of benzene (C_6H_6), which is the simplest and most abundant aromatic hydrocarbon in the solar system, have been extensively studied under pressure, the properties of benzene derivatives are poorly studied under extreme conditions.

In this work, we studied the pressure dependent structural properties of Phenylithium (C_6H_6Li) which is one of the simplest organo-alkali metallic compounds, in a diamond anvil cell for upto 32 GPa. We observed the initial polycrystalline lines under pressures of a few GPa. With the increasing of pressures, we have seen evidence of formation of new crystal structure under above 32 GPa with strong single crystal line. Phenylithuim has a strong luminescence under pressure which was evidenced by the changing color of the sample to orange. The structural studies have been done in advanced light source.

CP13. Swift Heavy Ion Irradiation of Dense GeO_2 Glass at High Pressure: Formation and Stabilization of a Disordered NiAs-Type Structure

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Research on materials under coupled extreme conditions including pressure, temperature, and irradiation has become a new and vibrant area of investigation [1]. In particular, ion-beams have been utilized to synthesize novel phases far from thermodynamic equilibrium [2]. The process involves the coupling of high pressure and large energy density deposition through the bombardment of pressurized samples by relativistic ions injected into a diamond-anvil cell. Most recently, we have applied the combined high-pressure and ion irradiation technique to amorphous germanium dioxide (GeO_2). The behavior of GeO_2 polymorphs under extreme conditions has been extensively studied due to the parallel in phase kinetics with the geologically- and technologically-significant silicon dioxide (SiO_2) system. Of particular interest in this study is the rare disordered niccolite (d-NiAs-type) structure of GeO_2. The d-NiAs-type structure of GeO_2 has previously only been formed through shockwave experiments and static experiments in a narrow temperature range (1000K-1300K) at pressures above 30 GPa [3]. Prior attempts to quench the phase were unsuccessful —noting a gradual transformation to stishovite within 20-60 hours. Here, we report on the crystallization and permanent stabilization of the (previously unquenchable) d-NiAs-type high-pressure structure of GeO_2 formed by *in situ* irradiation of amorphous GeO_2 with 35 GeV ^{197}Au ions at 47 GPa in the absence of high temperature. Preliminary XRD data of the quenched material illustrates a remnant amorphous phase with a modified signature that is potentially indicative of an amorphous-to-amorphous transformation.

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CP14. Irradiation-induced structural disordering on zirconate pyrochlores at high pressures

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The effects of swift heavy ion irradiation-induced disordering on zirconate pyrochlores ($A_2Zr_2O_7$ where A = Sm, Er, Nd) at high pressures are investigated using synchrotron x-ray diffraction. Irradiation experiments were performed at the GSI Helmholtz Center with ^{197}Au ions accelerated to energy of 2.2 GeV. Angle dispersive synchrotron powder x-ray diffraction measurements were completed at the Advanced Photon Source at Argonne National Laboratory, where samples were pressurized up to ~ 50 GPa using diamond anvil cells.

Analysis of the synchrotron x-ray diffraction data demonstrate that: (1) zirconate pyrochlores irradiated with swift heavy ions undergo high pressure phase transformations at >30GPa and the phase transformation is recoverable; (2) the irradiated high pressure phase for zirconate pyrochlores is a cotunnite-like phase; and (3) the disordering from ion irradiation increases the pressure stability range. The effects of disordering in different zirconate compositions under high pressure are explained in terms of the radius ratio of the cations.

CP15. High-Pressure, High-Temperature Equations of State Using Fabricated Controlled-Geometry Ni/SiO_2 Double Hot-Plate Samples

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To model and predict the structure, dynamics, and composition of Earth's deep interior, accurate and precise measurements of thermal expansion and compressibility are required. The laser-heated diamond-anvil cell (LHDAC) coupled with synchrotron-based x-ray diffraction (XRD) is a powerful tool to determine pressure-volume-temperature (*P-V-T*) relationships. However, LHDAC experiments may be hampered by non-uniform heating caused by the mixing of transparent materials with opaque laser absorbers. Additionally, radial temperature gradients are exacerbated by small misalignments (1-3 μm) of the x-ray beam with respect to the center of the laser-heated hotspot. We have fabricated three-dimensional, controlled-geometry, double hot-plate samples. In this double hot-plate arrangement, a transparent oxide layer (SiO_2) is sandwiched between two laser absorbing layers (Ni) in a single, cohesive sample. These samples were mass manufactured (>10^5 samples) using a combination of physical vapor deposition, photolithography, wet etching, and plasma etching. The double hot-plate arrangement coupled with the chemical and spatial homogeneity of the laser absorbing layers addresses problems caused by mixtures of transparent and opaque samples. The controlled-geometry samples have dimensions of 50 μm x 50 μm x 1.4 μm. The dimensions of the samples are much larger than the synchrotron x-ray beam. The radial temperature gradients within the volume probed by the x-ray are reduced when the radius of the laser-heating spot is ~25 μm. We conducted XRD experiments to P > 70 GPa and T > 2900 K at beamline 16-ID-B (HPCAT) of the Advanced Photon Source. Here we present relevant thermal modeling of the LHDAC environment along with Ni and SiO_2 *P-V-T* equations of state. Our photolithography method of sample fabrication can be extended to different materials including but not limited to Fe and MgO.

CP16. Synthesis and phase stability of Ni-Al-Co-Ti super alloys at high pressure using laser heating in DAC

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Cobalt based alloys have attracted broad attention due to their positive temperature dependence of strength and creep resistance better than commercial Ni based super alloys for the last 50 years. However, finding the stable L12 structured Co alloys were challenging. James et al 2011 [Advanced Materials Research Vol. 278 (2011) pp 399-404.] had recently reported a wide range of composition variation in the Ni-Al-Co-Ti system with L12 structure. However, there is no data available on the structural stability and mechanical properties of this system. we have synthesized Ni(70at%)-Al(15at%)-Co(12.35 at%)-Ti(2,65%) by arc melting followed by annealing. The microstructure and X-ray diffraction confirms the formation of the cuboids with space group Pm-3m (L12 type) structure in the Fm3m matrix. In the present work, the crystal structure stability at high pressures up to 20GPa studied using in-situ laser heating technique will be presented.

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CP17. Superconducting phase diagram of ^{6}Li: anomalous isotope effects in superconducting dense lithium

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We report the isotope effects in superconductivity of ^{6}Li and ^{7}Li samples between 16-26 GPa, measured simultaneously in a twin chamber gasket inside a diamond anvil cell (DAC). We find that below 21.5 GPa, Li exhibits a direct, but unusually large isotope effect, while above 21.5 GPa, the superconducting T_c of ^{6}Li falls below that of ^{7}Li, showing an inverse superconducting isotope effect; which has not previously been seen in any elemental superconductor other than α-U[1]. This effect remains present up to 26 GPa, the highest pressure studied here. Our results provide evidence that quantum solid effects dominate the low temperature properties of lithium under pressure.

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CP18. Phase transition and hydrogen bonding behavior in $Be(OH)_2$, $Zn(OH)_2$, and SiO_2 cristobalite at high temperatures and pressures

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Crystallographic comparisons of isotypical crystals and their responses under stress can describe how interatomic forces affect a crystal's structural stability, polymorphism and elasticity. This holds true for comparisons made with the SiO_2 system. Found both on the surface and in the interior of the Earth, SiO_2 exhibits complex behavior with modifications of pressure and temperature.[1] The variety of phase transitions and stability changes by silica can be attributed to its tetrahedral structure, along with the strong interactions between SiO_4 groups and weak Si-O-Si bonding.[2]

Many crystals with non-SiO_2-based compositions have SiO_2-like structures. One such isotypic family consists of behoite $Be(OH)_2$ and wulfingite $Zn(OH)_2$. These compounds are topologically identical to alpha-cristobalite; however, in the hydroxide crystals, the bridging oxygen atom has been replaced by a hydroxyl group. This introduces the effects of hydrogen bonding to the determination of structure and atomic parameters. These effects of hydration can be systematically studied using the SiO_2-$Be(OH)_2$-$Zn(OH)_2$ series.

For cristobalite, several phase transitions have been reported. A reversible change to the monoclinic cristobalite-II takes place at at 1.8 GPa [2], and was observed to be suppressed with a rapid increase in pressure.[3]An additional, non-reversible change to cristobalite X-I was observed at 12 GPa. The low-pressure transition of cristobalite is mirrored by behoite[3] and wulfingite[5], which both have phase transitions between 1.0 and 2.0 GPa, however, some of the characteristics in these hydroxide transitions are different, such as the magnitude of volume discontinuity. Behoite's high pressure phase was observed to be stable up to 10.4 GPa [3], with a very small discontinuity in volume. This could stem from how the hydrogen deforms the framework: additional hydrogen reduces the average Be-O-Be bond angle, further reducing the unit cell volume.[3] This stands in contrast to wulfingite, which experiences significant changes in crystal morphology and volume at the transitions.

Our work seeks to extend the information about the structural evolution of behoite and wulfingite with higher pressure and temperature, and their atomic displacement parameters. Additionally, earlier studies of high-pressure transformations in $Zn(OH)_2$ have not determined the exact structure - only unit cell and probable space groups.[5,6,7] We hope to use single crystal x-ray diffraction to determine the atomic positions and bonding in high-pressure $Zn(OH)_2$ crystal structures, and to make better comparisons against the behavior of behoite and cristobalite.

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CP19. Novel Radionuclide Wasteforms Prepared Under Pressure

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Currently, the most widely used waste form for nuclear waste is borosilicate glass. Although glass and ceramic waste forms have proven to be durable and sufficient at immobilizing many radionuclides; there are not ideal for certain radionuclides including I_2, Kr, Tc and actinides. These nuclear waste products have long half-lives and have particularly harmful health and environmental effects. There is a need to design new waste forms that can immobilize these problematic radionuclides and reliably store them for thousands – and in some cases millions of years. Zeolites are a family of either naturally occurring or synthetic aluminosilicate minerals composed of Earth-abundant, inexpensive, low toxicity elements. From observation of their natural analogs, they have shown to be stable in environmental conditions over long periods of time. Sodalite (a specific zeolite topology) is of particular interest because it is composed of a rigid framework of non-connected cavities (or cages). Once a guest molecule has been incorporated or inserted into a cage, it would be difficult for it to diffuse out. Although the diameter of the windows to these cages is smaller than the diameters of the intended guests; under temperature and pressure the windows can accommodate diffusion of larger guest molecules. Compression of the rhombohedral form of silica-sodalite was performed in hopes of learning the behavior and capabilities of the structure and how to possibly improve it for waste immobilization. Also, using General Utility Lattice Program (GULP) we predict pressure dependent changes to the structures.

CP20. Phase transformations of laser shocked NaCl with in situ X-ray Diffraction

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We have carried out dynamic compression experiments at the Matter in Extreme Conditions (MEC) endstation at the Linac Coherent Light Source, Stanford National Accelerator Laboratory. The combination of nanosecond laser drive and high brightness x-rays with ~50 fs pulse durations allow the in situ material response to dynamic compression to be investigated.

Samples consisted of single-crystal NaCl, a thin Au or Al pressure calibrant, and a polyimide ablator. Laser energies at 527nm were as low as 5 J (one beam), with a maximum possible energy of ~26 J (two beams overlapped) contained within a 200-400µm focal spot. Targets were compressed to pressures of up to 80 GPa over 10 ns duration, while simultaneous velocity interferometry measured the particle velocity history and the timing of shockwave breakout from both the Au/Al layer and the free surface. By varying the delay of the x-ray pulse with respect to the laser pulse, in situ x-ray diffraction patterns capture the sample crystal structure before, during and after shock wave propagation through the sample. Diffracted x-rays were collected on megapixel x-ray cameras (CSPADs) positioned downstream from the shock target.

The evolution of NaCl structure as a function of time allows us to track its response through shock compression to different high pressure states and subsequent relaxation to ambient pressure. Direct comparison between measured structure and density to simulation of shock propagation and equation of state allows the kinetic response to dynamic compression to be studied.

CP21. Single-crystal Brillouin spectroscopy with laser-heating and variable q

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We present a Brillouin spectroscopy system integrated with CO_2 laser heating and Raman spectroscopic capabilities. High-pressure laser-heating experiments have been performed on liquid water compressed in a diamond-anvil cell. Temperature was determined by measurements of the grey-body thermal radiation emitted by the hot sample, with the system response calibrated relative to a standard tungsten ribbon lamp. Brillouin measurements of acoustic velocities on liquid water and ice were performed at elevated temperatures up to 2500 ± 150 K at high pressure. The ability to perform Raman measurements with the same system was demonstrated. Single-crystal laser-heating Brillouin measurements were made on the (111) plane of San Carlos Olivine at ~13 GPa, 1300 ± 200 K. The pressure as measured by ruby fluorescence is shown to be close to the pressure on the olivine sample during laser-heating when KCl and KBr are used as a pressure-transmitting media. In addition, the system is designed for continuously varying scattering angles from near forward scattering (0º scattering angle) up to near back scattering (~141º). This novel setup allows us to probe a wide range of wave vectors **q** for investigation of phonon dispersion on crystals with large unit cells (on the scale of hundreds of nm).

CP22. T-Rax and Dioptas: new Python-based programs for on-the-fly optical spectroscopy and 2D X-ray diffraction data analysis

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T-Rax and Dioptas are open source Python programs with easy to use graphical user interfaces and powerful analysis tools. T-Rax handles wide range of spectroscopic data collected with Princeton Instr. detectors (WinSpec or LighField file format) including thermal radiation, fluorescence and Raman spectroscopy. Temperature of laser or externally heated samples is calculated by fitting grey-body radiation curve to collected spectra with optional correction of the known system response. Unique features are automatic new file processing, fast switching between different system response calibrations and options, graphical selectable regions of interest, time-series analysis and communication with EPICS. Furthermore, T-Rax can calculate pressure at various T from Ruby spectra, with optional peak fitting, and Diamond spectra, with showing the derivative of the spectrum.

Dioptas is intended to be a modern improvement of the widely used FIT2D software (Hammersley, 1997). It uses the pyFAI (Kiefer & Wright, 2013) library as its basis for integration of 2D X-ray diffraction images and provides uniquely designed and easy to use interface for image auto-processing, browsing, calibration of any detector geometry and mask creation. The mask widget includes undo/redo options and an automatic cosmic ray removal procedure. All parameters can be saved and reloaded. MAR-CCD and Perkin Elmer 2D images (2048 x 2048 Pixel) could be integrated within 50ms or less (FIT2d takes a few seconds). Integrated spectra window provides the option of displaying and manipulating with unlimited overlays. The positions of individual diffraction lines of selected phases are shown and can be adjusted to pressure and temperature when the appropriate equation of state parameters are given.

T-Rax has started being used for user operation at the GSECARS station in September, 2013, and Dioptas has been successfully employed for user operation since June 2014.

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Workshop on high-pressure time-resolved synchrotron techniques

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