HPCAT Project Narrative

May 2016

Executive Summary

The High Pressure Collaborative Access Team (HPCAT) project is to advance high-pressure science and technology in multidisciplinary fields using high energy and high brilliance synchrotron radiation. HPCAT operates the Sector 16 of the Advanced Photon Source (APS) of Argonne National Laboratory, where an array of high-pressure x-ray diffraction, x-ray spectroscopy, and x-ray imaging techniques have been developed and established for users' operation in four simultaneously operational beamlines.

The HPCAT project is managed by Carnegie Institution of Washington. The project was initiated in 1999, followed by the construction in 2000, and the first beam received in the experimental hutch in late 2002. Since then, the HPCAT facility has been open for users' operations, and has been exceedingly successful in advancing high-pressure science and technology. The operation of HPCAT is currently supported by DOE-NNSA under Award No. DE-NA0001974 and DOE-BES under Award No. DE-FG02-99ER45775, with partial instrumentation funding by NSF.

As a fundamental thermodynamic variable, pressure drastically and categorically alters all chemical, structural, mechanical, electronic, magnetic, and phonon properties, and pushes materials across boundaries between insulator, semiconductor, metal, and superconductor, ionic, metallic, and covalent bonding, vigorously reactive and inert elements, amorphous and crystalline solids, etc. Surprising high-pressure physics and chemistry are revealed and novel materials are created. In search of materials with extreme properties for energy and environment technology, pressure presents a vast unexplored, fertile ground in which "unexpected" scientific discoveries are the norm, and accelerated advances and breakthroughs are expected. High-pressure materials are not merely for academic interests. Numerous examples at HPCAT have demonstrated that a great number of novel phases and their superior properties discovered under high pressures can be preserved and recovered after releasing pressure (quenched) for applications at the ambient condition. The discoveries at high pressure also give crucial clues for designing alternative chemical routes to synthesize the advanced materials. Furthermore, pressure provides a clean knob for tuning materials properties that reveals in-depth understanding of novel phenomena.

To investigate physical and chemical properties in-situ under high pressures, we need to have powerful micro-sampling probes to reach the minute samples through the walls of the pressure vessels and to distinguish the weak sample signals from the often overwhelming background signals produced/excited by the massive surrounding vessel materials. High-brilliance, high-energy, low-emittance synchrotron is a perfect solution and is unleashing the full potential of high-pressure science. At HPCAT, a plethora of synchrotron x-ray techniques has been developed and integrated with high-pressure experimentations for understanding the atomic, electronic, and magnetic structures and their relationships to materials properties under pressure. The *high-pressure x-ray diffraction techniques* at HPCAT yield comprehensive crystallographic information, including atomic structure, bonding, density, elasticity, deviatoric strain, rheology, and preferred orientation of single-crystal, polycrystalline, nanocrystalline, and non-crystalline materials in-situ under high pressures and at high/low temperatures. The *high-pressure x-ray spectroscopy* techniques at HPCAT include *x-ray emission spectroscopy* which provides information on the filled electronic states of the compressed samples; *x-ray Raman spectroscopy* which reveals pressure-induced chemical bonding changes of the light

elements; *inelastic x-ray scattering spectroscopy* which accesses the high-energy electronic phenomena, including electronic band structure, Fermi surface, excitons, plasmons, and their dispersions; *resonant inelastic x-ray scattering spectroscopy* which probes shallow core excitations, multiplet structures, and spin-resolved electronic structure; and *nuclear resonant x-ray spectroscopy* which provides phonon densities of state and time-resolved Mössbauer information. The *high-pressure x-ray imaging* techniques clearly resolve the size, shape and textural changes induced by pressures and temperatures. These x-ray probes integrated with hydrostatic or uniaxial compression, laser heating, and cryogenic cooling, have enabled investigations of structural, vibrational, electronic, and magnetic properties that were unimaginable only a decade ago.

More than 230 individual users on average have performed experiments at HPCAT in each of the past three years. Many are partner users and returning users, making the "person-visits" more than 570 each year. Among them, more than 60% are students and post-doctoral associates, reflecting a critical role of HPCAT in training next generation scientists by providing the state-of-the-art platform to broad user communities. Every year, there are about 10 Ph.D. and Master degree theses completed based fully or in part on experiments performed at HPCAT. In addition to numerous reports to the federal programs, HPCAT experiments have resulted in more than 2 peer reviewed papers per week, among which >14% appear in high profile journals with impact factors $\geqslant$ Physical Review Letters.

On the horizon of the Upgrade of the Advanced Photon Source (APS-U), together with the new developments in high-pressure technologies, a new era is emerging in high-pressure science, one that revolutionarily redefines the scope of high-pressure research and promises an in-depth understanding of matter under extreme conditions across the entire hierarchy of length and time scales. The HPCAT Upgrade (HPCAT-U) will meet these new challenges in high-pressure science and technology with the new opportunities. Recently, a new scope of the APS-U has been approved in 2015 and the APS-U project has been developing very fast since then. The APS-U will provide a 100 to 1000 times increase in brightness and coherence at high energy that will dramatically improve the ability to study smaller samples with finer spatial and temporal resolution with unparalleled precision and accuracy. Moreover, new developments in high-pressure technology (e.g., double stage diamond anvils, rapid compression or decompression, pulsed or modulated laser heating) raise new demands for novel synchrotron techniques with orders of magnitude improvements in spatial and temporal resolution. New developments in synchrotron techniques, together with advanced detectors and high power computing algorithms, provide unprecedented opportunities for probing matter under extreme conditions. The HPCAT upgrade aims at grasping these opportunities to keep HPCAT facility at the leading edge, and will accomplish three goals: (1) advancing to the next generation high-pressure synchrotron science, (2) enabling new techniques and capabilities by taking the full advantage of the APS-U with unmatched brilliance and coherence, and (3) further strengthening the HPCAT role as a central pipeline for training next generation scientists by providing the state-of-the-art platform to broad user communities.

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1. The HPCAT Project

1.1 HPCAT – an integrated synchrotron facility for advancing high-pressure science and technology

Supported by DOE-NNSA and DOE-BES grants, the High Pressure Collaborative Access Team (HPCAT) operates the sector 16 of the Advanced Photon Source (APS) of Argonne National Laboratory dedicated for high pressure research. HPCAT facility has taken advantage of the nation's most brilliant high energy synchrotron source and developed a multitude of integrated synchrotron radiation (SR) techniques optimized for advancing high-pressure (HP) science and technology. These x-ray probes, integrated with hydrostatic or uniaxial compression, static or dynamic loading, resistive or laser heating, and cryogenic cooling, have enabled users' investigations of structural, vibrational, electronic, and magnetic properties at HP and high/low temperatures that were not possible a decade ago. New discoveries and observations of new phenomena become the norm in daily operation of this vibrant facility. Research work at HPCAT provides fundamental knowledge of the behavior of materials in a broad range of environments, such as pressure, temperature, radiation, and deviatoric stress, including both static and dynamic phenomena. Investigations of structure, equations of state, and electronic and magnetic properties provide critical data for code validation and tests of fundamental theory. The measured "structure-property" correlations help to establish predictive models for developments of new materials and new applications. Data on materials properties at conditions occurring within the Earth and planetary interiors provides essential keys for understanding the mineralogy, dynamics and composition of the Earth and other planets.

Figure 1: HPCAT: an integrated high pressure facility located at Sector 16 of the Advanced Photon Source.

1.2 History, organization, and facility staff

HPCAT started in 1999 with a DOE-BES Grant to the Carnegie Institution of Washington (CIW) and a one-time CIW-endowment seed fund. Soon afterwards, DOE-NNSA funded Lawrence Livermore National Laboratory (LLNL) and University of Nevada at Las Vegas (UNLV) to join as HPCAT Members, and several years later, University of Hawaii (UH, a 5% Member supported by a DOD grant, discontinued after 3 years) and the DOE-NNSA funded Carnegie/DOE Alliance Center (CDAC) also joined as HPCAT Members. Before 2013, HPCAT operation was supported by a consortium of four Members (CIW, CDAC, UNLV, and the Tri-Labs of LLNL and LANL). Since 2013, HPCAT operation is supported by two DOE grants to CIW (*PI-Shen*), with one from DOE-NNSA supporting at 75% level, and the other from DOE-BES at 25% level.

The HPCAT project is managed by CIW, which is also an institutional partner. Other institution partners include CDAC, UNLV, and Tri-Labs (LLNL, LANL, SNL); the research programs in these institution

partners are supported by DOE-NNSA. The designated representatives of the managing partner (CIW) and other stakeholders (CDAC, UNLV, LLNL, LANL) constitute the Executive Council (EC) which serves as the primary governing body of HPCAT. The managing partner appoints an Executive Director of the Council, presiding over the EC, and a Director of HPCAT, supervising the HPCAT project. An external Advisory Committee (AC) meets annually and reviews strategic directions to maintain HPCAT as a forefront facility serving scientific interests of the partners and missions of the funding sponsors. The AC report in 2015 is included in the Appendix 5.1.

Onsite staff of the HPCAT facility:

Director	Guoyin Shen
Associate Director	Stanislav Sinogeikin
Beamline Scientists	Arunkumar Bommannavar (Software Control) Paul Chow Yoshio Kono Yue Meng Changyong Park Dmitry Popov Jesse Smith Yuming Xiao
Beamline Associates	Daijo Ikuta Curtis Kenney-Benson
Support Staff	Freda Humble (Project Administrator) Eric Rod (Beamline Technician)
Postdoctoral Associates	Ross Hrubiak Chuanlong Lin Cheng Ji, 100% supported by DOE-BES Jianjun Ying, 100% supported by DOE-BES Bingyang Hou, supported by a research grant

Members of the Executive Council:

Managing partner - Carnegie Institution of Washington

- Executive Director (*V. Struzhkin*)
- HPCAT Director (*G. Shen/S. Sinogeikin*)

DOE-NNSA partners (S. Brown/K. LeChien/R. Schneider)

- CDAC (*R. Hemley/S. Gramsch*)
- HiPSEC-UNLV (*A. Cornelius*)
- NNSA Tri-Labs
  - LLNL (*W. Evans/H. Cynn*)
  - LANL (*N. Velisavljevic*)
  - SNL (*C. Seagle*)

DOE-BES partner (L. Wilson)

- CIW (*G. Shen/H.K. Mao/ V. Struzhkin*)

1.3 Guidelines for HPCAT developments

In the development of HP-SR science, we consider three important characteristics. First, HP is a vast multidisciplinary field impacting physics, chemistry, materials, geosciences, and biosciences. Secondly,

HP research is made much more powerful by integrating other dimensions of extreme environments, i.e., extreme temperature, extreme magnetic field, extreme radiation flux, extreme strain rate, etc., which requires considerable expertise and effort. Thirdly, each individual high energy SR technique coupled with a HP sample is a valuable probe for a specific property, but the combination of several techniques form an extremely powerful tool for addressing major questions. Based on these characteristics, we use the following four guidelines in the development of HPCAT facility:

- *Science driven* – the techniques are chosen for their maximum impact in the multidisciplinary and interdisciplinary HP sciences.
- *Optimized for HP devices* - the x-ray energy, energy resolution, x-ray beam size, background discrimination, detector configuration, etc. are optimized with consideration of HP vessels and samples at HP conditions.
- *Integrated capabilities* - the multiple techniques at HPCAT are integrated for complementary information to enhance the common scientific goal.
- *Comprehensive support equipment* - a number of supporting instruments have been developed for controls and measurements of pressures and temperatures, complementary optical and magnetic measurements, and sample handling and preparation.

1.4 HPCAT beam time and user community

HPCAT Beam time is allocated according to a system that meets the following guidelines.

- Only high quality proposals are eligible for allocations of beam time.
- 25% of the beam time are reserved for general users, as required by APS.
- Up to 8% may be allocated for beamline maintenance and machine study.
- Up to 7% may be allocated for beamline development projects.
- Up to 10% may be allocated for specific technical developments or for scientific projects proposed by HPCAT Director or partners.
- All remaining beam time, with a minimum of 50%, shall be distributed among the partners, in proportion to their interests specified by the funding sponsors. The current beam time distribution percentages are as follows:

  DOE-NNSA partners (75%) → CDAC (30%), NNSA Tri-lab (25%), UNLV (20%)

  DOE-BES partners (25%) → CIW

General users may access the HPCAT beam time by submitting general user proposals (GUPs) through a peer-reviewed system managed by APS. HPCAT beam time is highly oversubscribed among the GUPs.

HPCAT institutional partners receive guaranteed beam time at HPCAT with percentages listed above.

UNLV: With support from DOE-NNSA, the UNLV High Pressure Science & Engineering Center (HiPSEC) brings together chemists, geoscientists, engineers, and physicists to study materials properties under extreme conditions. HiPSEC users of the HPCAT facility include 10 faculty members, more

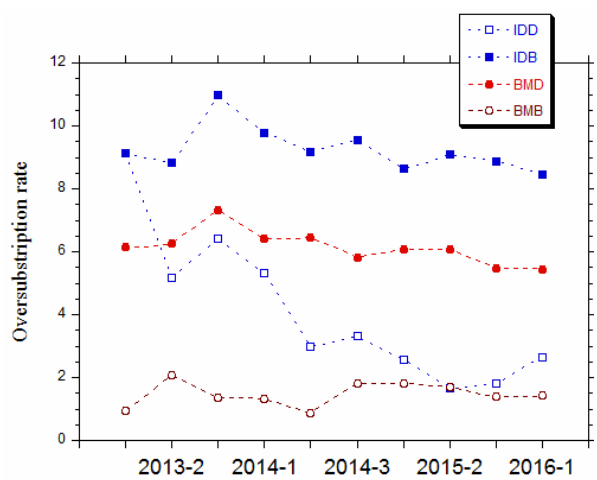


Figure 2. Oversubscription rate at HPCAT beamlines according to general users' proposals. The rate is calculated as the numbers of shifts requested over the allocated beamtime.

than 10 postdocs, an average of 15 graduate students, and a number of undergraduate students.

Tri-lab: The Tri-lab groups includes scientists from the three major DOE-NNSA sponsored national laboratories: Livermore (LLNL), Los Alamos (LANL), and Sandia (SNL). The HP groups in these laboratories consists of more than 30 staff scientists and postdocs focusing on the scientific challenges of condensed matter and synthesis and characterization of novel materials under extreme P-T conditions. Materials of particular interest include *f*-electron lanthanides and actinides, *d*-band transition-element metals and compounds, low-Z elemental and molecular solids, and novel superhard and high energy-density materials of scientific and DOE-NNSA programmatic importance.

CDAC is funded by the Stewardship Sciences Academic Alliance Program of DOE-NNSA to advance an extensive set of high P-T techniques and unique facilities, to perform key studies on a comprehensive array of materials in newly-accessible P-T regimes, to integrate static, dynamic and theoretical results for stewardship science, and to build the HP community in the U.S. CDAC distributes its HPCAT beam time to more than 50 users among its 15 academic partners in universities and in national laboratories. CDAC itself represents a large portion of U.S. high-pressure community.

CIW is one of the world's leading institute in HP research. The CIW HP research group has been engaging in the development of a multitude of HP-SR techniques at HPCAT and conducting research in all aforementioned HP scientific areas.

1.5 HPCAT user and publications

HPCAT is dedicated for high pressure research, serving the user community in the U.S. and beyond. The community is multidisciplinary in physics, chemistry, materials science, Earth and planetary sciences, and biological sciences. HPCAT has made significant effort in communicating with the community to (1) promote several standardized designs of HP device for the community, (2) make universal adapters for accommodating various types of devices, and (3) develop new designs for expanding the extent of extreme conditions or performing new types of experiments. In addition to the diamond anvil cell device, we have employed a large volume press (Paris Edinburgh cell) for comprehensive studies of liquids at high pressures.

More than 230 individual users performed experiments at HPCAT in each of the past three years (see for example a list of individual users in 2015 in Appendix 5.4). Many are partner users and returning users, making the “person-visits” more than 570 each year. Among them, more than 60% are students and post-doctoral associates. In average, there are >10 Ph.D. theses and >3 master degree theses completed based partly on experiments at HPCAT every year. In addition to numerous reports to the DOE programs, HPCAT experiments result in >2 peer reviewed papers per week, among which >14% appear in high profile journals with impact factors ≥ Physical Review Letters/Impact factor: 7.7.

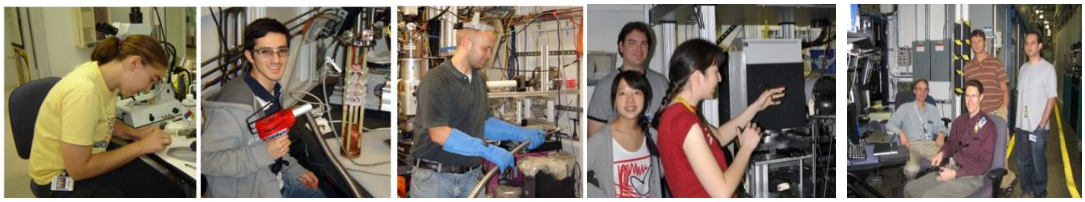


Figure 3: More than 230 individual users each year performed experiments at HPCAT. Among them, more than 60% are students and post-doctoral researchers.

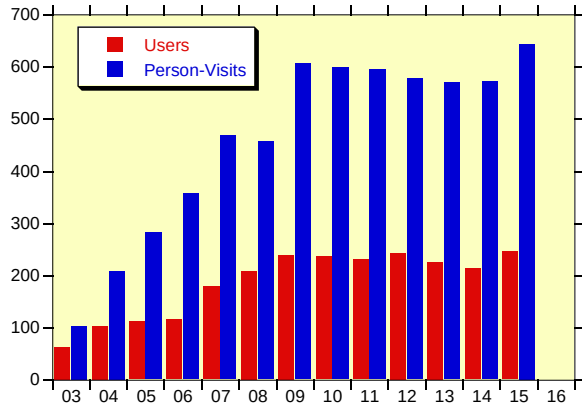


Figure 4. More than 230 individual users come to HPCAT every year. Counting return users, the "person-visits" are more than 570 every year.

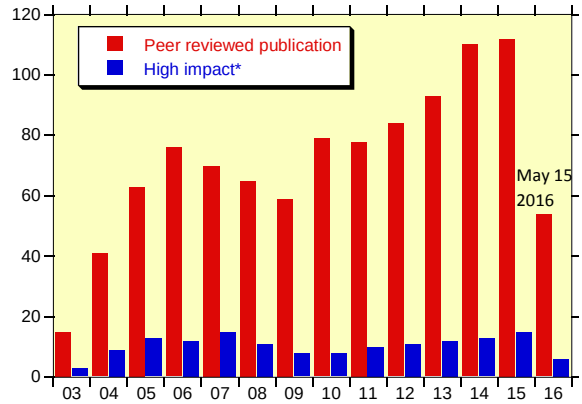


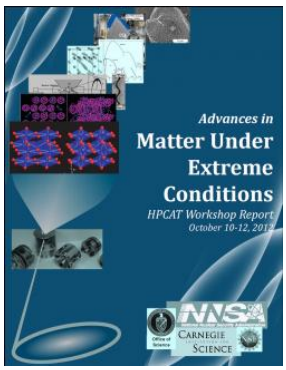
Figure 5. HPCAT is one of the most productive sectors at APS. More than 2 peer-reviewed papers are published every week, with a number of them appearing in high profile journals.

In the "End-of-Experiment" surveys managed by APS, we receive very positive feedback (1 or 2 on a scale of 1-5 with 1 being the best) from users. See the users' feedback information in 2014 and 2015 in the Appendix 5.5. Occasionally, when we receive a score of 4 or worse in certain areas, HPCAT management will follow up and address the issues in a timely manner.

1.6 Workshops, conferences, and outreach

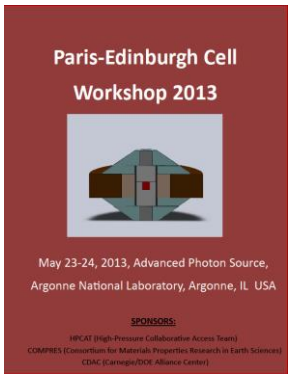
Efforts are made to ensure the HPCAT facility is responsive to the needs of the HPCAT partners and the scientific users' community, and that the community is made aware of the capabilities and new developments at the facility. Some examples of the outreach efforts that we have undertaken include:

Workshops or short courses: In average, we organize at least one workshop or short course on HP synchrotron science and technology each year. After the "***HPCAT Workshop on Matters under Extreme Conditions***" in October 10-12, 2012 at the APS, which was to celebrate the 10th anniversary of HPCAT operation and to discuss the priority research directions in HP science, we have organized several more focused /specific workshops.



- **A *Paris-Edinburgh Cell Workshop*** was held on May 23-24, 2013. The workshop consisted of four technical training courses as well as a discussion forum related to the current status of Paris-Edinburgh cell applications in HP research worldwide. The workshop presented an opportunity for practical hands-on training to a total of 32 graduate students, post-docs, as well as young and senior researchers among the participants, who were totally new or only partially experienced to the Paris-Edinburgh cell applications.
- A Workshop on ***high-pressure time-resolved synchrotron techniques*** was held on September 25-27, 2014. More than 100 participants attended this two-and-a-half-day workshop. The workshop focused on novel and emerging techniques and capabilities, and the presentations were mostly given by renowned experts in the related fields in order to provide a balanced representation and coverage in specific sub-fields. The

attendees represented a diverse group of researchers at various career stages from different countries. Attendance of 32 young career scientists (students and postdocs) was made possible through generous travel grants from COMPRES and CDAC.



- A workshop on ***HPCAT computer and software support*** was held on January 29, 2015. Discussion topics included HPCAT network configuration, EPICS basics, and available software packages for equipment control and support of experimental techniques at HPCAT beamlines. Discussions also covered the beamline software requirements for quick and meaningful data reduction to assess the quality of the data during runtime.
- HPCAT held a special workshop on ***highest static pressure*** on May 6, 2015. More than 25 scientists have reviewed the historic landmarks and recent experiments in which the highest static pressure was achieved (or claimed to have been achieved) in Diamond Anvil Cells (DAC) (above 400 GPa) and in large volume presses (LVP) (above ~100 GPa). Special attention has been paid on the rapidly developing double-stage anvil cell techniques and developments. Extensive discussions of the next steps for achieving the highest possible static pressure in DAC and LVP have been made.

High Pressure Special Interest Group: HPCAT organizes a special interest group on HP techniques and synchrotron applications at the APS. This group consists of more than 30 people at Argonne National Laboratory, from nearby universities, including users on site; the group meets monthly.

High-pressure Synchrotron Discussion Forum: This is a monthly discussion forum since 2010. One of the staff selects a topic of interest for discussion in the area of HP synchrotron science and technology. The forum helps to keep all HPCAT staff at the frontiers in HP synchrotron research.

HPCAT Postdoc / Student / Visitor talk series: Starting in March 2015, every HPCAT post-doc, student, or long-term visitor makes a presentation about his/her research at HPCAT and/or beyond. This talk series serves as a very important educational platform preparing young scientists for professional development in academic environment. The talks are typically given once a month depending on the current number of HPCAT postdocs, students, and visitors.

Argonne Neutron and X-ray Summer School: HPCAT has participated in this two-week school since 2004. Students perform hands-on experiments using the micro-diffraction equipment at HPCAT beamlines.

Attending Meetings and Visiting Other Synchrotron facilities: HPCAT staff are often invited to present research and development at various workshops and meetings. We also regularly (at least one time per year per scientific staff) attend scientific meetings to present new results and/or new capabilities. We invite people from other synchrotron facilities to visit HPCAT and HPCAT staff also visit their facilities.

In addition to these workshops, conferences, and regular discussion forums, HPCAT often hosts visitors from local high schools and universities nationwide. For example, we often host groups of *Science Careers in Search of Women* and summer students from UNLV.

1.7 HPCAT project budget

The HPCAT operation is supported by two DOE grants, with a DOE-NNSA grant (DE-NA0001974) supporting at 75% level, and a DOE-BES grant (DE-FG02-99ER45775) at 25% level. The current funding period for NNSA grant is 5 years from January 1, 2013 to December 31, 2017, while the BES grant is for 3 years from September 1, 2015 to August 31, 2018. Over the years, HPCAT has received NSF-MRI support through CIW for major instrument procurements or upgrades such as monochromators and detectors. The HPCAT budget in 2015 was $3,511,361, with its spending categories shown in Fig. 6.

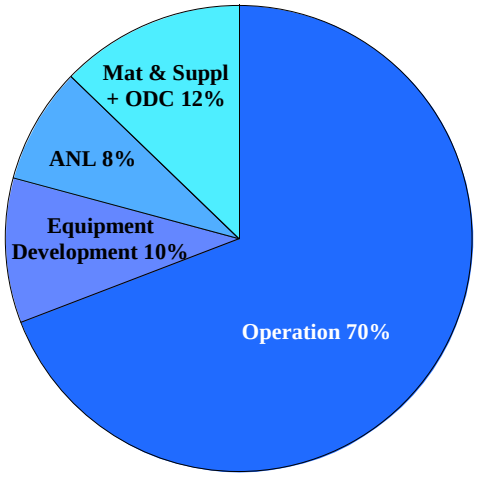


Figure 6. Spending categories of HPCAT budget.

2. HPCAT Experimental Capabilities

2.1 HPCAT beamlines

Dedicated for HP research, an array of x-ray techniques in diffraction, spectroscopy, and imaging have been developed or pioneered at HPCAT for addressing fundamental questions in physics, chemistry, materials sciences, geosciences, and biosciences. There are four simultaneously operational beamlines at HPCAT (16-ID-B, 16-ID-C/D/E, 16-BM-B, 16-BM-D), with specialized x-ray optics and HP-SR instruments in nine stations.

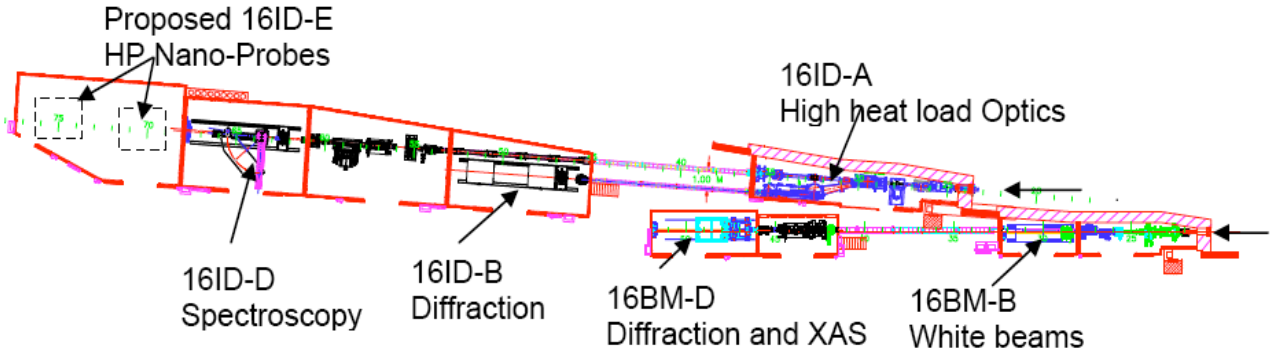
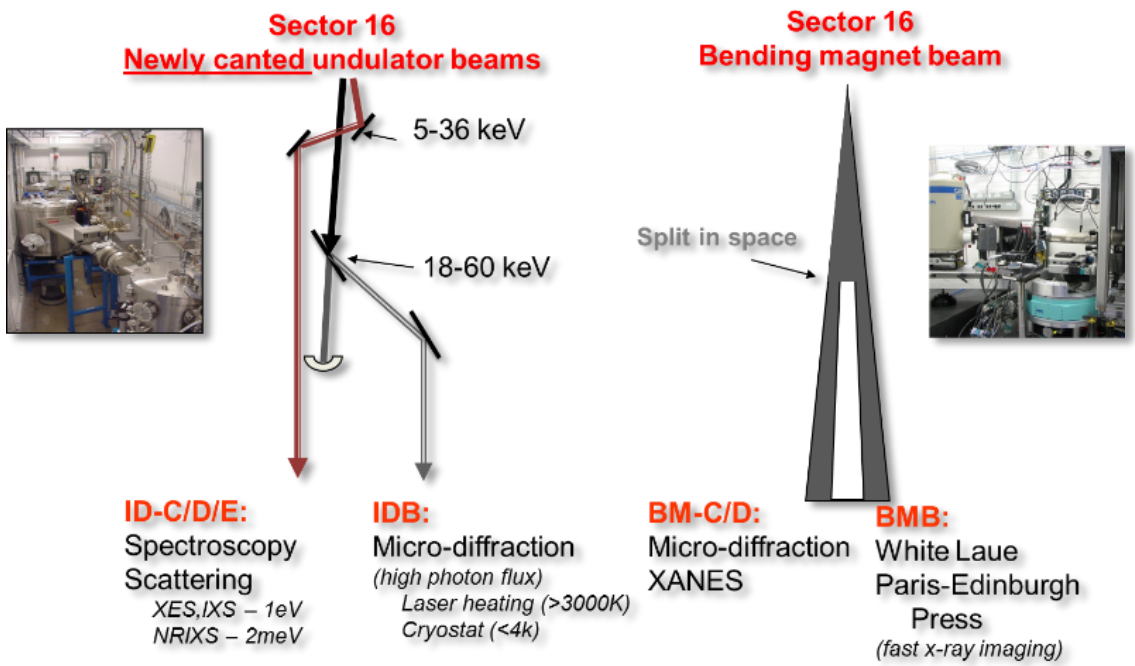


Figure 7: HPCAT sector layout. The locations of four simultaneously operational stations are marked.

To increase the efficiency in utilization of precious beam time, the 16ID and 16BM beamlines are each split into two branches (Fig. 7). The canted undulators allow for establishing two independent ID beamlines: the 16ID-C-D-E branch for a range of HP x-ray spectroscopy techniques and the 16ID-B branch for HP x-ray diffraction (XRD). The white radiation fan of 16BM is divided into two halves: one into 16BM-B for HP energy dispersive x-ray diffraction (EDXD) and Laue diffraction, and one into 16BM-C-D for HP micro XRD and x-ray absorption spectroscopy (XAS).



2.2 Established HP-SR techniques

An array of synchrotron x-ray techniques has been developed and integrated with high-pressure experimentations for understanding of atomic, electronic, and magnetic structures and their relationships to materials properties under extreme pressures and temperatures. The **HP x-ray diffraction techniques** at HPCAT yield comprehensive crystallographic information, including atomic structure, bonding, density, elasticity, deviatoric strain, rheology, and preferred orientation of single-crystal, polycrystalline, nanocrystalline, and non-crystalline materials in-situ under HPs and at high/low temperatures. The **HP x-ray spectroscopy techniques** at HPCAT include *x-ray emission spectroscopy* which provides information on the filled electronic states of the compressed samples; *x-ray Raman spectroscopy* which reveals pressure-induced chemical bonding changes of the light elements; *inelastic x-ray scattering spectroscopy* which accesses the high-energy electronic phenomena, including electronic band structure, Fermi surface, excitons, plasmons, and their dispersions; *resonant inelastic x-ray scattering spectroscopy* which probes shallow core excitations, multiplet structures, and spin-resolved electronic structure; and *nuclear resonant x-ray spectroscopy* which provides phonon densities of state and time-resolved Mössbauer information. The **HP x-ray imaging techniques** resolve size, shape and textural changes induced by pressures and temperatures. These tools integrated with hydrostatic or uniaxial compression, laser and resistive heating, as well as cryogenic cooling, have enabled investigations of structural, vibrational, electronic, and magnetic properties that were unimaginable only a decade ago. A collection of 10 papers was recently published in a single volume, vol. 86 July 2015, of the Review of Scientific Instruments. These papers represent a snapshot of recent developments at HPCAT in advancing HP-SR techniques.

The following ten sections describe the established capabilities, the unique features, and enabling scientific programs at HPCAT beamlines. Follow the links (control + click) for more details.

2.2.1 HP x-ray diffraction – undulator beamlines
2.2.2 HP x-ray diffraction – bending magnet beamlines
2.2.3 HP in situ x-ray diffraction with laser heated diamond anvil cell
2.2.4 HP x-ray Laue diffraction
2.2.5 HP inelastic x-ray scattering
2.2.6 HP x-ray emission spectroscopy
2.2.7 HP nuclear scattering
2.2.8 Paris-Edinburgh press facility
2.2.9 Laboratories and support equipment
2.2.10 Beamline controls and data management

3. Advances in High Pressure Science

HPCAT is in the harvesting stage, reflected by the advancement and flourishing achievements in HP-SR science. The following examples highlight the magnitude and breadth of recent advances since 2013 in multidisciplinary HP-SR science enabled by the HPCAT facility [1-375]. Student and postdoc research is emphasized in selecting these examples to stress the educational aspect of HPCAT.

3.1 Exotic "simple" elements

• Diversity of the carbon family and the Group IV elements

Nanoarchitectured carbon: HPCAT experiments showed that at pressures above 40 GPa, the arrangement of the bonds between the C atoms in the glassy carbon completely shift from sp^2 to sp^3 that produces the spheres with diamond-like strength while disordered structure of the spheres remains unchanged, thus representing a new carbon allotrope. Searching for new carbon allotropes with unusual physical properties has long been a subject of extensive studies. The recent work on nano-architectured carbon exhibits unusual and tunable mechanical properties, similar to mechanical metamaterials but with basic length units at nanometer scale. These results show that such combined forms can inherit the distinct properties of individual allotropes and thus possess exceptional physical properties [319].

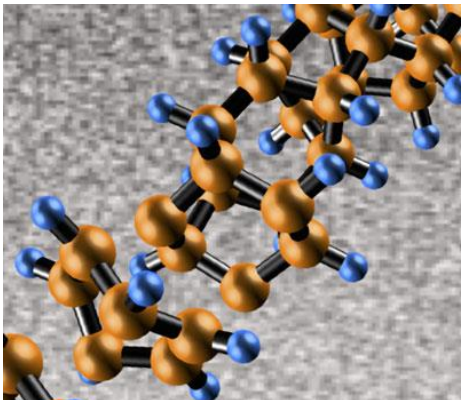


Figure 8. 'Diamond nanothreads' promise extraordinary properties, including strength and stiffness greater than that of today's strongest nanotubes and polymers.

Diamond nanothreads: One-dimensional sp^3 carbon nanomaterials, recoverable to ambient pressure, can be formed by HP solid state reaction of benzene (Fig. 8). They form in close packed bundles of nano-carbon threads capped with hydrogen. These nano threads promise extraordinary properties such as strength and stiffness higher than that of sp^2 carbon nanotubes and polymers. The core of the nanothreads is a long, thin strand of carbon atoms arranged just like the fundamental unit of a diamond's structure – zig-zag "cyclohexane" rings of six carbon atoms bound together, in which each carbon is surrounded by others in the strong triangular-pyramid shape of a tetrahedron [236].

An open-framework allotrope of silicon: In order for silicon to be more attractive for use in novel energy technologies, its indirect band gap needs to be altered. A new form of silicon has been synthesized (Fig. 9) with a quasi-direct band gap that falls within the desired range for solar absorption, something that has never before been achieved. Unlike the conventional diamond structure, this new allotrope consists of an interesting zeolite-type structure, which is comprised of channels with five-, six-and eight-membered silicon rings. The new compound was obtained using an innovative high-pressure precursor process, and is recoverable to ambient pressure [252].

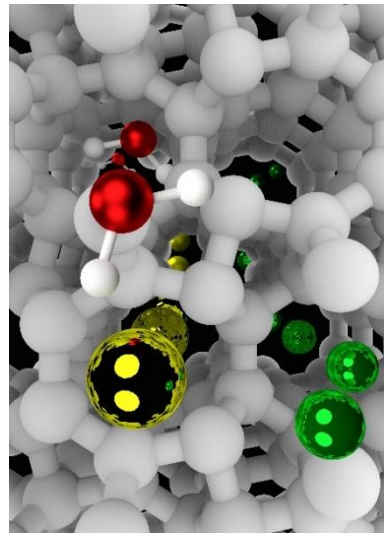


Figure 9. The new Si_{24} allotrope has an open-framework structure comprised of 5-, 6- and 8-membered sp^3-bonded silicon rings.

- Novel phases of nitrogen

A layered polymeric nitrogen phase: A novel phase of nitrogen at pressures and temperatures exceeding 125 GPa and 3000 K (well above the synthesis conditions of the polymeric cubic gauche phase) have yielded a single-bonded, layered polymeric nitrogen (LP-N) phase with a remarkable structure and extraordinary properties. The new structure is characterized by 3D (cg-N) to 2D transition to the theoretically predicted Pba2 structure, consisting of seven-membered N-N rings [179].

A new λ-N_2 phase showing stability in 1-140 GPa: A novel phase of nitrogen, λ-N_2, has been synthesized through low temperature compression. The new phase exhibits an exceptionally wide range of pressure stability from below 1 to 140 GPa, overlapping with nine other known phases. The P-T conditions under which λ-N_2 is in thermodynamic equilibrium are unknown and pose an interesting challenge for future theoretical studies [330].

- *Chain breakage in liquid sulfur*: HPCAT's high-energy x-rays were utilized to study the local order of liquid sulfur at HP-HT conditions. A temperature driven structure change in liquid sulfur was observed, signified by an order of magnitude reduction in lengths of sulfur chains. The chain breakage may strongly influence the physical properties, such as the semiconductor-metal transition and a drastic decrease in viscosity across the transition [147].

3.2 *f*-electron metals and their alloys

Evidence of liquid-liquid transition in cerium: A pressure-volume isotherm in cerium metal at 1100 K was measured in a large volume press of the Paris-Edinburgh type up to 6 GPa (Fig. 10). The data show a pronounced decrease of the bulk modulus above the gamma-phase region similar to the 775 K isotherm in the solid that also shows an inflection point between gamma and alpha-type cerium [144].

Strength and Debye temperature of cerium have been obtained by directly measuring the sound velocities of polycrystalline samples across the $\gamma \rightarrow \alpha$ volume collapse [50].

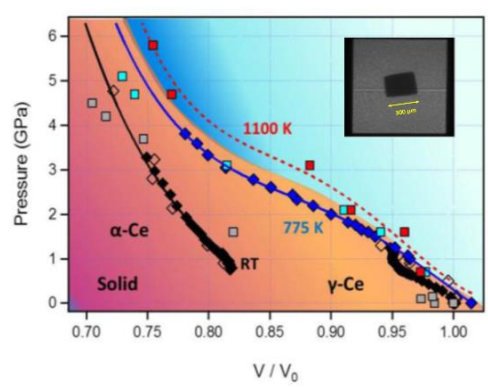


Figure 10. Isothermal p-V compression data of Ce. Insert: a sample sitting inside a cell assembly as imaged by x-ray beam.

Phase diagram of gadolinium has been mapped using a boron-doped designer heater anvil. Meanwhile, equations of state of each phases (hcp, αSm, dhcp) have been measured using the in situ x-ray diffraction technique [347].

Elasticity and thermal properties of depleted uranium have been determined using ultrasonic interferometry coupled with the Paris-Edinburgh cell at HPCAT [335]. This work has also demonstrated the capacity to make such measurements on radioactive and other potentially hazardous materials.

Equations of state for technetium: The compression data shows that the HCP phase of technetium is stable up to 67 GPa. The result shows an excellent agreement between X-ray diffraction and DFT calculations [346].

Magnetism in uranium sulfide: Under pressure, the Curie temperature of uranium sulfide is reduced monotonically until it discontinuously disappears near a pressure-induced deformation of the crystal structure. This HP distortion is identical to the one correlated with the onset of magnetic order, but with a larger change in the cell angle [30].

Intermediate valence state in the Kondo insulator SmB_6: Applied pressure reduces the *f*-electron occupancy, but surprisingly, the material maintains a significant divalent character up to a pressure of at least 35 GPa (Fig. 11). Over the entire pressure range, the material maintains a remarkably stable intermediate valence that can in principle support a nontrivial band structure [323].

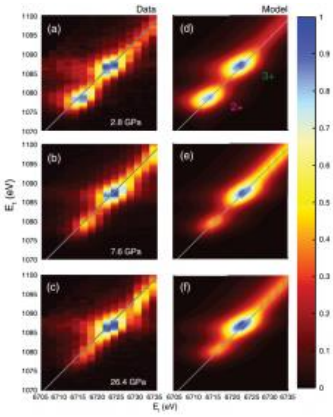


Figure 11. Comparison of resonant x-ray emission data (a)–(c) and theoretical fits (d)–(f) at different pressures. With increasing pressure, the lower energy peak associated with the divalent state is suppressed.

Coupled electronic, magnetic, and lattice degrees of freedom in $LaCo_5$: Coupled degrees of freedom are at the root of the emergent behaviors of functional materials. Ferromagnetic $LaCo_5$ shows an anisotropic lattice collapse of the c axis near 10 GPa that is also commensurate with a change in the majority charge carriers evident from HP Hall effect measurements [294].

Partial fluorescence yield (PFY) reveals delocalization of 5f electrons in UCd_{11}: Under compression, the PFY measurements show that the white absorption line is shifted by +4.1(3) eV at HP indicating delocalization of the 5*f* electrons. The increase in full width at half maxima and decrease in relative amplitude suggest 6*d* band broadening under HP [348].

Redox response of actinide materials to highly ionizing radiation: Energetic radiation can cause dramatic changes in the physical and chemical properties of actinide materials, degrading their performance in fission-based energy systems. Oxidation state of actinide ion caused by swift heavy ion radiation is coupled with structural distortion, which generates similar effects caused by Frenkel defect in the crystalline structure (Fig. 12). The studied materials include ThO_2, CeO_2, and UO_3, from which the direct comparison reveals that ThO_2 has distinct

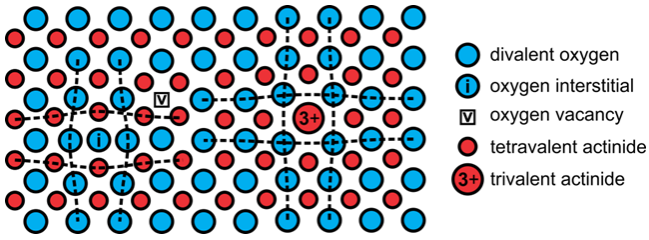


Figure 12. Illustration of the effect of the oxidation state reduction of tetravalent actinide to trivalent state causing a lattice distortion.

damage accumulation process relative to other two cases because thorium is stable only in a tetravalent state. The radiation tolerance of these materials depends on the efficiency of the redox reaction, such that damage can be inhibited by altering grain size and cation valence variability [180,299].

3.3 Novel superconductivity

Pressure-decoupled magnetic and structural transitions of $BaFe_2As_2$: By combining synchrotron Mössbauer spectroscopy and X-ray diffraction, magnetic and structural transitions of the parent compound of iron-based 122 superconductors $BaFe_2As_2$ have been studied under HP and low temperature conditions, showing that the magnetic ordering transition precedes the structural transition in $BaFe_2Fe_2$ [82] (Fig. 13).

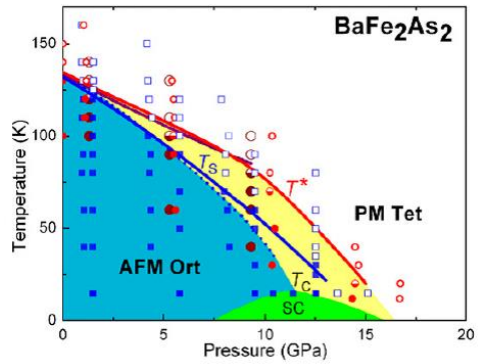


Figure 13. The magnetic ordering transition precedes the structural transition in $BaFe_2Fe_2$.

Superconductivity in highly disordered dense carbon disulfide: The superconductivity in molecular systems is extremely rare. Usually, but not always, superconductivity is present in highly ordered molecular structures but, counter to the understanding of how superconductivity normally works, in carbon disulfide the superconductivity arises from a highly disordered extended state (CS_4 phase or phase III[CS_4]) at ~6.2 K over a broad pressure range from 50 to 172 GPa [10].

Superconductivity stabilized by electron-hole interband coupling: The appearance of a distinct superconducting phase above 13 GPa is found to be accompanied by a sudden reversal of dominant charge carrier sign, from hole- to electron-like. The high-temperature superconducting phase in KFe_2As_2 is substantially enhanced by the presence of nested electron and hole pockets, providing the key ingredient of high-Tc superconductivity in iron pnictide superconductors [275].

Pressure-induced superconductivity in a topological material $ZrTe_5$: The T_c is found to increase with applied pressure, and reaches a maximum of 4.0 K at 14.6 GPa. At pressures above 21.2 GPa, a second superconducting phase with the maximum T_c of about 6.0 K appears. The observed two-stage superconducting behavior is correlated to the structural phase transition from ambient Cmcm phase to HP C2/m phase around 6 GPa, and to a mixture of two HP phases of C2/m and P-1 above 20 GPa [375].

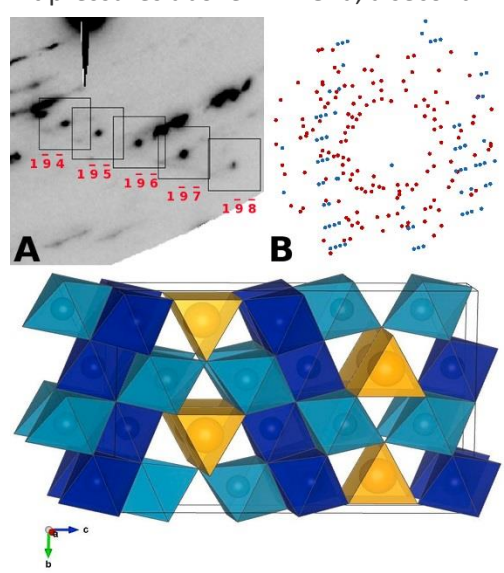


Figure 14. The structure of Fe_5O_6.

3.4 Rule breaking chemical compounds and chemical stability at HP

Unexpected stable stoichiometries: New materials with different stoichiometries emerge at HP even for very common compound, such as a cubic and orthorhombic $NaCl_3$ and a two-dimensional metallic tetragonal Na_3Cl at high pressures [88].

Novel iron oxides: The peculiar properties of iron oxides triggered scientific curiosity for thousand years. In spite of extensive investigation, only three compounds have been

reported so far: FeO, Fe_2O_3, Fe_3O_4. HPCAT experiments revealed several new compounds, Fe_4O_5, Fe_5O_6, which are found to be retrievable at ambient conditions. We are revealing an unforeseen complexity in the Fe-O system with four different compounds—FeO, Fe_5O_6, Fe_4O_5, and *h*-Fe_3O_4—in a narrow compositional range (0.75 < Fe/O < 1.0) [258].

Novel xenon chemistry: A novel HP compound, $Xe(H_2)_8$, with the highest hydrogen content of all known compounds was recently synthesized [291]. The synchrotron x-ray diffraction and Raman measurements show that this unique hydrogen-bearing compound that can be synthesized at 4.2 GPa and 300 K, is retrievable at low temperatures to atmospheric pressure, and retained up to 90 K on subsequent warming.

Exotic, stable calcium carbides: A recent systematic search for stable calcium carbides was carried out using evolutionary structure prediction calculations. From ambient pressure up to 100 GPa, six calcium carbide compounds were predicted to be stable with one or more crystal structures throughout the explored pressure range. Researchers used HPCAT to successfully synthesize and subsequently characterize two of these predicted compounds, Ca_2C and Ca_2C_3, which demonstrate the powerful synergy between computational and experimental studies [261].

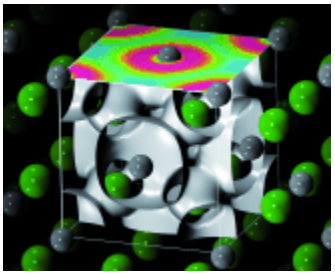


Figure 15. Normalized electron density distribution in Mg_2C.

Ionic carbon: When magnesium and carbon combine in a 2:1 ratio above 15 GPa, a new antifluorite structure is formed (Fig. 15). The compound, with composition Mg_2C, is highly ionic, with carbon in a very unusual C4− methanide state [38].

Polymeric CO via useful hard X-ray photochemistry: When a strontium oxalate (SrC_2O_4) is irradiated by high energy synchrotron radiation at HP, a dark reddish/brown region formed in the area of irradiation which was surrounded by a yellowish brown remainder in the rest of the sample. Further analysis suggests that the sample is polymerized CO with dispersed CO_2 and SrO trapped within the polymer [349].

Polymerization of tetracyanoethylene (TCNE): TCNE in a quasi-hydrostatic condition undergoes a shear-induced phase transition at 10 GPa and then a chemical change to two-dimensional (2D) C=N polymers above 14 GPa. These phase and chemical transformations depend strongly on the state of stress in the sample and occur sluggishly in non-hydrostatic conditions over a large pressure range between 7 and 14 GPa. Laser-heating of the C=N polymer above 25 GPa further converts to a theoretically predicted 3D C–N network structure [79].

Polymorphism of Tetrahydroxy-p-benzoquinone: A HP phase is observed above 11.5 GPa for tetrahydroxy-p-benzoquinone. The high-pressure phase is highly photosensitive and easily polymerized [324].

Phase stability of 2,4,6-trinitrotoluene (TNT): TNT is a molecular explosive that exhibits chemical stability in the molten phase at ambient pressure. At HP, the chemical stability of molten TNT is limited, existing in a small domain of pressure-temperature conditions below 2 GPa. Decomposition dominates the phase diagram at high temperatures beyond 6 GPa. From the calculated bulk temperature rise, it is unlikely that TNT melts on its principal Hugoniot [105] (Fig. 16). In crystalline phase, while Raman

spectroscopy measurements indicate spectral changes at ~2 GPa, careful XRD measurements reveal that the monoclinic phase persists up to 10 GPa [98].

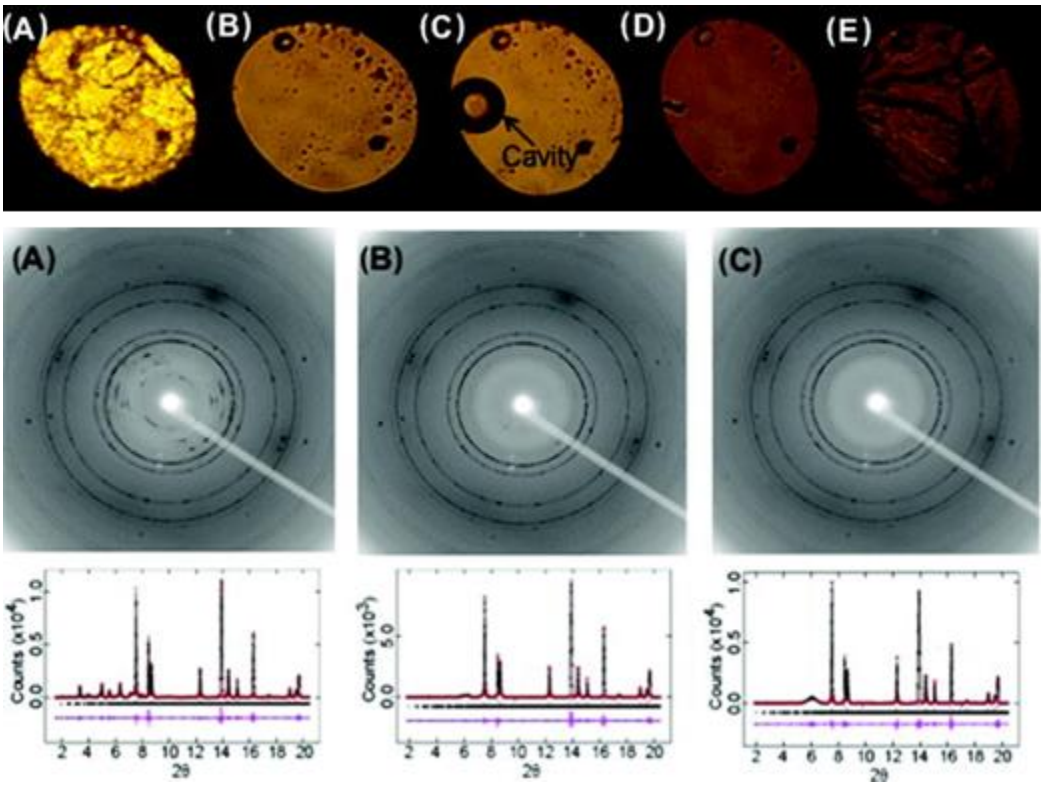


Figure 16. (Top) Photomicrographs of TNT in the sample chamber of a DAC, illustrating the melt/recrystallization behavior of TNT. (Bottom) X-ray diffraction patterns of TNT showing the progression from crystalline phase to decomposition.

3.5 HP glasses and liquids, polyamorphism

Fractal short-range and homogeneous long-range structures in metallic glasses: Metallic glasses earn their name from a lack of long-range atomic order and the absence of typical defects, such as dislocations, and exhibit exotic material properties. Recent HPCAT experiments revealed a crossover between fractal short-range (<2 atomic diameters) and homogeneous long-range structures in metallic glasses [224]. The fractal short-range framework is consistent with the general 2.5 power law observed for density of metallic glasses. Interestingly, this general power law even persists across the first-order polyamorphic transition of a $Ce_{68}Al_{10}Cu_{20}Co_2$ metallic glass under HP. As this transition is equivalent to a compositional change from 4*f*-localized Ce (big) to 4*f*-itinerant Ce (small), the result signifies the commonality of the fractal dimension over wide variety of compositions and density of metallic glasses [370] (Fig. 17).

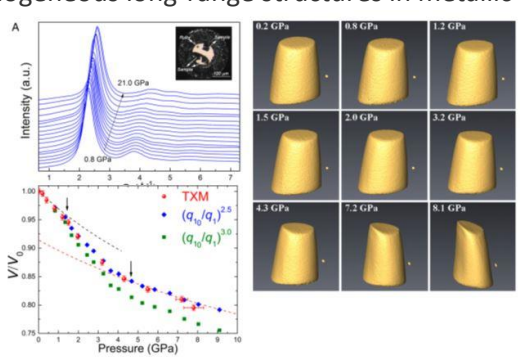


Figure 17. (left) In-situ high-pressure XRD measurements of $Ce_{68}Al_{10}Cu_{20}Co_2$ metallic glass from 0.8 to 21.0 GPa. (right) Three-dimensional reconstruction of tomographic data of transmission X-ray microscopy at different pressures.

How does structure influence viscosities and densities of silicate melts at high pressures? The degree of polymerization is a defining characteristic of silicate melt,

and it affects the properties of silicate melts (e.g., viscosity, density). Structural analyses on silicate melts ($NaAlSi_2O_6$-$CaMgSi_2O_6$ join) at HP show that structures of polymerized and depolymerized melts respond to pressure in distinct manner, resulting in different viscosity and density behavior. The viscosity turnover in polymerized liquids corresponds to the tetrahedral packing limit, below which the structure is compressed through tightening of the inter-tetrahedral bond angle and continual break up of tetrahedral connectivity. Above the turnover pressure, silicon and aluminum coordination increases to allow further packing [191].

Anomaly in viscosity correlates with structural changes in ionic compounds: Viscosity is one of the most fundamental transport properties in liquid. Recent HPCAT development of high-speed x-ray radiography combined with a Paris-Edinburgh cell enabled viscosity measurements of low viscos (<1 mPa s) liquids and fluids. A falling sphere technique revealed an anomaly in the viscosity of liquid KCl at around 2 GPa. Structural data of liquid KCl showed a pronounced change signified by the ratio r2/r1, where r1 and r2 are the nearest- and the second-neighbor distances, respectively. The results suggest that the viscosity anomaly in liquid KCl strongly correlates with the structural changes [36].

Ultrahigh pressure polyamorphism in GeO_2 glass: A new double-stage large volume cell has been developed at HPCAT to compress GeO_2 glass samples to nearly 100 GPa for in situ structure measurements (Fig. 18). The experiments revealed new evidence of ultrahigh pressure polyamorphism in GeO_2 glass with Ge-O coordination number (CN) significantly greater than 6. The CN change is closely associated with the change in oxygen packing fraction [340].

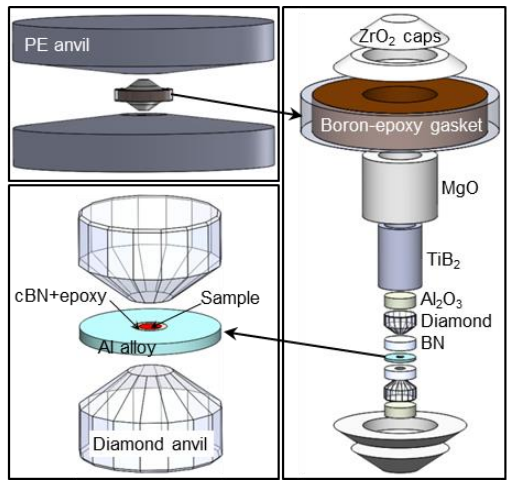


Figure 18. A double stage configuration for megabar experiments with a large volume press.

3.6 Kinetics and metastability

Phase transition kinetics: Under various compression rates (0.03–13.5 GPa/s), kinetics of the B1-B2 phase transition in KCl has been studied in a dynamic diamond anvil cell using time-resolved x-ray diffraction and fast imaging. The volume fraction across the transition generally gives sigmoidal curves as a function of pressure during rapid compression. Based upon classical nucleation and growth theories (Johnson-Mehl-Avrami-Kolmogorov theories), the fit of the experimental volume fraction as a function of pressure provides information on effective activation energy and average activation volume at a given compression rate. The resulting parameters are successfully used for interpreting several experimental observables that are compression-rate dependent, such as the transition time, grain size, and over-pressurization [341].

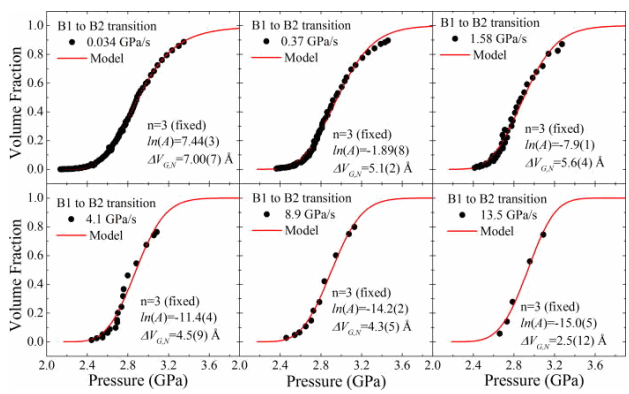


Figure 19. Volume fraction of the KCL-B2 phase as a function of pressure at various compression rates. Black circles are the experimental data. Red lines are the fits from the JMAK model with n fixed at 3.

Kinetics of the $\alpha-\omega$ transition in zirconium: Using DAC-XRD time resolved measurements, the kinetics of the α to ω transformation in Zr was investigated. A clear time and pressure dependence is observed in the martensitic α-ω transition as a function of compression rate. There is likely no intermediate phase occurring between α and ω phases, with no evidence of the HP β phase [247].

Structural intermediates of compressed coesite: HPCAT experiments revealed that compressing single-crystal coesite SiO_2 under hydrostatic pressures of 26–53 GPa at room temperature results in a new polymorphic phase transition featured by the formation of multiple previously unknown triclinic phases of SiO_2 on the transition pathway as structural intermediates. These intermediate phases are accompanied by extensive splitting of the original coesite X-ray diffraction peaks, appearing as dramatic peak broadening and weakening, resembling an amorphous material. This work provides new insights into the pressure induced amorphization and the densification mechanism of tetrahedrally coordinated structures [243].

Metastable phases of silicon and germanium: While some of HP polymorphs (such as many silicates) can be kinetically recovered upon pressure release, there are many HP phases that are not retrievable, and transform to low P phases with decreasing P. Under rapid decompression process, however, very interesting and useful metastable phases can be synthesized. Various paths have been explored at HPCAT to recover the desirable metastable phases for silicon and germanium with excellent photovoltaic properties [117,240].

3.7 HP materials in environmental and Earth sciences

Understanding Earth's ultralow velocity zones: Near the Earth's core-mantle boundary (CMB) there occur isolated regions characterized by anomalously high density and low seismic wave velocity. The non-uniform distribution of these so-called ultralow velocity zones (ULVZs) is evidence of chemical and/or thermal heterogeneity near the CMB. Recent experiments at HPCAT suggest the eutectic melting curve crosses the geotherm near the CMB, and thus it is possible that regions of dense metallic melt, originating from slab material in the lower mantle, form near the CMB [343] (Fig. 20).

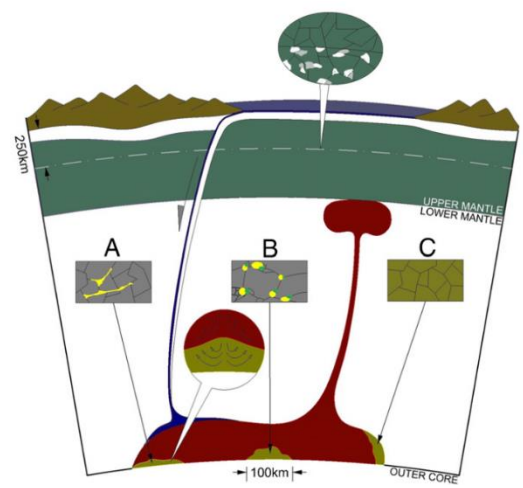


Figure 20. Illustration depicting three types of ULVZs involving slab-derived iron-carbon melts.

Iron isotopes constrain core chemistry: The separation of iron metal from silicate to form Earth's core represents a fundamental physical and chemical differentiation process in our planet's history. Isotope fractionation exists between phases with distinct bonding environments (e.g., Earth's core and mantle), and separation of elements between reservoirs manifests this fractionation. Recent HPCAT experiments using nuclear forward scattering technique show the effect of pressure on iron isotopic composition, which are found to vary according to the alloy tested (FeO, FeH_x, or Fe_3C). These results suggest that hydrogen or carbon is not the primary light-element component in the core [353].

Hidden carbon in the Earth's core: Certain seismic waves move slower than expected through the Earth's core, causing researchers to rethink what the innermost region of our planet is made of. New research on a form of iron-carbide, Fe_7C_3, shows that it may have the required low seismic wave velocity

at high pressure. The speed of seismic waves in an iron-carbide, Fe_7C_3, has been measured at core conditions, suggesting that the iron-carbide's anomalous velocity behavior is due to a change in the electron spin configuration of iron in the material. The results could imply that the Earth's core is rich in Fe_7C_3 [102].

Ultralow viscosity of carbonate melts: Falling sphere viscosity measurement using ultrafast X-ray imaging in Paris-Edinburgh cell at 16-BM-B, HPCAT, revealed that viscosities of carbonate (calcite and dolomite) melts are surprisingly low; in the range of 0.006-0.010 Pa s. These low viscosities are close to that of water and are orders of magnitude lower than those of silicate melts. This implies that the mobility of carbonate melts (defined as the ratio of melt-solid density contrast to melt viscosity) is ~2-3 orders of magnitude higher than that of basaltic melts [130].

Irreversible Xe insertion into a zeolite at subsurface Earth condition: It has been known that the Earth's atmosphere is depleted in Xe by a factor of 20 relative to the lighter noble gases – neon, argon, and kryption, which is referred as 'missing xenon paradox'. Recent HPCAT experiments shed a new light on this problem (Fig. 21). A zeolite, Ag-natrolite, can absorb and retain 28 wt.% of Xe at 1.7 GPa and 250 ˚C, a condition found in subsurface Earth. The oxidation state change in Ag, disproportionation of Ag^+ into Ag^0 and Ag^{2+}, is observed during the insertion and desorption processes, which indicates a possible stabilization of the Xe compound [170].

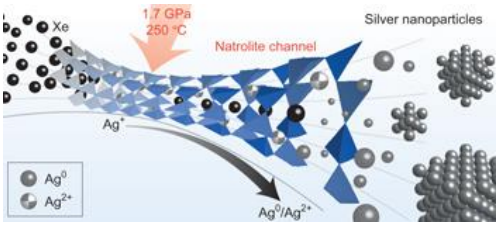


Figure 21. Illustration of Xe insertion into Ag-natrolite zeolite under a high pressure and temperature condition and the accompanying Ag^+ disproportionation into Ag^0 and Ag^{2+}.

Lower mantle chemistry breakthrough: Previous models of a perovskite-dominant lower mantle have been built on the assumption that the entire lower mantle down to the top of the D" layer contains ferromagnesian silicate [$(Mg,Fe)SiO_3$] with nominally 10 mole percent Fe. On the basis of experiments at HPCAT using laser-heated DAC, at 95 to 101 GPa and temperatures of 2200 to 2400 K, it is found that such perovskite (Pv) is unstable; it loses its Fe and disproportionates to a nearly Fe-free $MgSiO_3$ perovskite phase and an Fe-rich phase with a hexagonal structure called the H phase. This finding suggests that $(Mg,Fe)SiO_3$ Pv may not be the major silicate throughout the lower mantle down to the top of the D″ layer. For the lower mantle beyond ~2000 kilometers depth, all geodynamic models may need to be reconsidered to take the H-phase into account [203].

4. HPCAT Upgrade Opportunities

A new era is on the horizon in HP science, one that revolutionarily redefines the scope of HP research and promises an in-depth understanding of matter under extreme conditions across the entire hierarchy of length and time scales. The HPCAT Upgrade (HPCAT-U) is to meet these new challenges in HP science and technology in the light of the new opportunities including the on-going Upgrade of the Advanced Photon Source (APS-U) and novel HP synchrotron techniques that have recently become feasible and practical.

The HPCAT-U will be in line with the APS-U project. As soon as the APS-U resumes its operation after the major lattice reconfiguration in 2021-2022, all HPCAT beamlines will resume operation immediately. Meanwhile, we will undertake the commissioning of the planned enabling techniques and capabilities, and make 75% of them available to users within the first year after the operation is resumed. The remaining 25%, mainly related to new x-ray imaging techniques, will be open to users in the next 2-3 years after the completion on the APS-U upgrade. These enabling techniques and capabilities will

provide the superior tools for the user community for bringing the development of HP science to the next level. The upgrade of HPCAT is essential and necessary to meet these new challenges, will take the full advantages of the new opportunities, and will keep the HPCAT at the frontier in the HP synchrotron research worldwide. Furthermore, the enabling state-of-the-art platform with the upgrade for advancing fundamental knowledge of matter under extreme conditions will strengthen the HPCAT role as a central pipeline for training next generation scientists.

4.1 Challenges and opportunities

The HPCAT program has been exceedingly successful in scientific impact, technological advances, and user community development. Fourteen year's explorations at HPCAT have revealed clear directions for the next generation of HP synchrotron research. Our prototype facility, developed more than a decade ago, is aging and facing steep competitions from newly established HP beamlines in Europe and Asia that largely adopted the HPCAT innovations. Meanwhile, a new scope of the APS-U has recently been approved in 2015 and the APS-U project has been developing very fast since then. The APS-U will provide a 100 to 1000 times increase in x-ray brightness and coherence at high energy and that will dramatically improve the ability to study smaller samples with finer spatial and temporal resolution and greater precision and accuracy. Moreover, new developments in HP technology (e.g., double stage diamond anvils, rapid compression or decompression, pulsed or modulated laser heating) raise new demands of novel HP synchrotron techniques with orders of magnitude improvements in spatial and temporal resolution. New developments in synchrotron techniques, together with advanced detectors and high power computing algorithms, provide unprecedented opportunities for probing matter under extreme conditions. Important questions arise, such as:

- What next generation HP science will these new developments enable?
- How can new enabling capabilities be applied for experiments at higher pressures, by both static and dynamic devices, and more extreme temperatures than previously possible with nanometer spatial resolution and sub-nanosecond temporal resolution?
- How can we benefit from the much improved spatial resolution to study hierarchical structures from the bonding, atomic, molecular, to nanoscale levels for understanding their intriguing bulk macroscopic behavior at extreme conditions?
- How can we use the much improved temporal resolution, applied over the rapid compression (decompression) and/or modulated heating (cooling) processes, for understanding how phases are nucleated, their growth, and non-equilibrium transformations to define their dynamical properties of bulk materials?

The HPCAT upgrade aims at grasping these unprecedented opportunities to keep the facility at the leading edge, and will accomplish three goals: (1) advancing to the next generation HP synchrotron science, (2) enabling new techniques and capabilities by taking the full advantage of the APS-U with unmatched brilliance and coherence, and (3) further strengthening the HPCAT role as a central pipeline for training next generation scientists by providing the state-of-the-art platform to broad user communities.

4.2 The upgrade of the Advanced Photon Source (APS-U)

The Advanced Photon Source is the brightest storage ring synchrotron radiation source in the nation in the hard x-ray range. It is presently in the midst of an upgrade to deliver even higher performance and more powerful capabilities for scientific research. A new avenue for creating high brightness x-ray beams has been recently accepted in 2015, based on the use of multi-bend achromat (MBA) magnet lattices in the storage ring. With the new MBA lattice, planned to be operational in 2021-

2022, the emittance in horizontal direction will be significantly reduced (Fig. 22), resulting in a more "round" x-ray beam ideal for developing powerful new capabilities for scientific research.

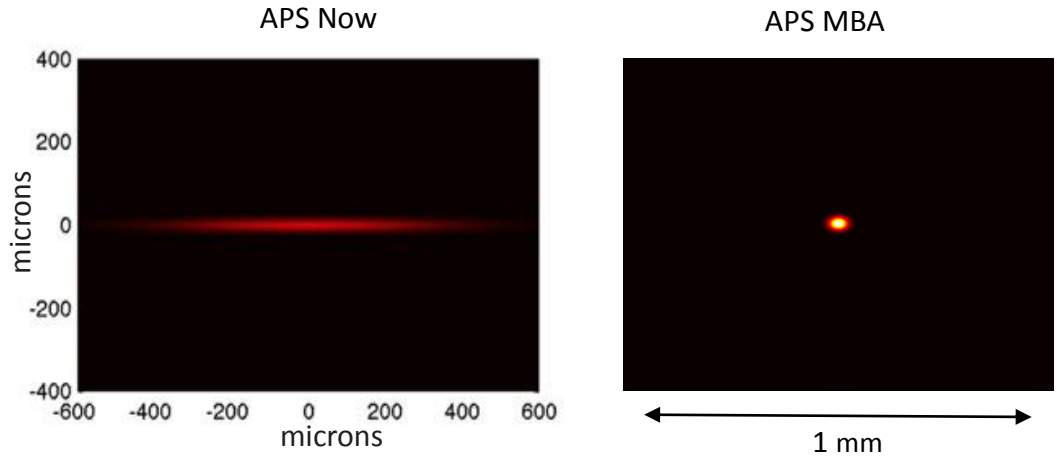


Figure 22. The horizontal emittance will be significantly reduced in the APS-U, delivering a much brighter and more coherent round-shaped beam.

The MBA lattice will dramatically reduce the emittance of the APS storage ring, and the use of advanced undulators, such as superconducting undulators, and other novel insertion devices, such as three-pole wiggler, offers unprecedented performance in x-ray production from the low-emittance electron beam. The APS-U will provide a 2-3 orders of magnitude increase in brightness and coherence at high energy. The low emittance and high brightness of the APS-U source is well suited for HP-SR research, and will dramatically improve the ability to study smaller samples and faster processes with greater precision and accuracy. The exceptionally high coherence fraction will allow new coherence based x-ray imaging techniques to be developed for 3-dimensional morphology and strain distributions of high pressure samples, and for studies of the slow dynamics of various equilibrium and non-equilibrium processes at high pressure. As Dr. Dean Haeffner, the Experimental Facility Associate Project Manager in the APS-U, said "the MBA lattice in the APS-U is most beneficial to, and almost tailored for, high pressure synchrotron research". To fully realize these exciting developments, HPCAT must similarly be capable of operating with the highest possible performance.

4.3 Scientific objectives of HPCAT-U

We have engaged the broad scientific community who have come together to look forward to the opportunities and research directions for HPCAT and for HP science communities in general. These ideas have been explored through several workshops held at HPCAT in 2012, 2013, and 2014. In the HPCAT-U we will focus on five directions:

- redefining the scope of HP research by extending pressures beyond 0.5 TPa
- revolutionary understanding of complex materials across the entire hierarchy of length scales and heterogeneity of materials at HP
- studying kinetics, pathways, and metastability under rapid (de)compression and pulsed (or modulated) laser heating at HP
- accurately probing phase transitions and chemical reactions at megabar pressures

- high throughput characterization probes for exploiting extreme environments for materials design, pathways, and synthesis

4.4 Enabling techniques and capabilities

The source enhancement with the APS-U provides great opportunities for developing next-generation x-ray probes for HP research. To ensure that HPCAT beamlines being capable of operating with the highest possible performance, the HPCAT-U project will focus on developing several enabling techniques with submicron spatial resolution and submicro-second temporal resolution that take the most benefit from the source upgrade in the APS-U and integrate these techniques for studying more complex materials, smaller samples, and faster processes with greater precision and accuracy. Some examples include:

- enabling sub-micron x-ray probes integrated with HP devices
- orders of magnitude improvements in spatial and temporal resolution
- advanced x-ray imaging techniques for complex hierarchical structures
- in situ measurements relating microscopic structures and macroscopic properties
- bridging the strain rate gap between static and dynamic compression

A full proposal for the HPCAT-U project will be submitted in summer 2016.

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The year of 2013:

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6. APPENDICES

6.1 HPCAT Experimental Capabilities
6.2 2015 Report of the HPCAT Advisory Committee
6.3 2013 (Latest) Review Report by the Advanced Photon Source
6.4 List of Individual Users in 2015
6.5 Users' Feedback After Experiments in 2014 and 2015

APPENDIX 6.1: HPCAT experimental capabilities

6.1.1 HP x-ray diffraction – undulator beamlines [back]

The experimental station 16-ID-B, one of two insertion device beamlines at HPCAT, is a dedicated beamline for monochromatic, angle-dispersive HP micro-XRD studies of materials contained in DAC. The experimental hutch includes two distinct end-stations: a laser heating table optimized for in-situ LH DAC experiments (described in more detail below), and a general purpose table designed to accommodate a wide variety of HP experimental needs, measurement apparatus, and supporting equipment. 16-ID-B offers exceptionally high photon flux, a broad energy range, tightly focused beam, high performance sample motion and positioning, and various types of large-format x-ray area detectors. 16-ID-B makes it possible to study the structure and properties of materials at ultrahigh pressures up to several hundred GPa, extreme temperatures from below 4K up to several thousand K, and more recently, over a broad range of time scales down to sub-millisecond [290].

The undulator radiation source for 16-ID-B offers continuous energy coverage and high flux at relatively high energies (~18-60 keV) particularly relevant to DAC XRD. The experimental x-ray wavelength is selected using a liquid nitrogen cooled side-bounce double-crystal monochromator which includes three pairs of Si crystals – (111), (220), and (311) – offering a choice among highest flux, greatest flexibility, or largest Q-range. Recent developments include the installation of a 2.6 m long granite air bearing monorail for precise positioning of the second monochromator crystal. This helps to ensure stable and reliable beam delivery and to facilitate rapid energy changes depending on users' experimental needs. Near-future developments include the installation of a diamond window beam position monitor offering real-time, state-of-the-art monitoring and control of the beam position and intensity during user experiments.

Focusing is achieved using one of two Kirkpatrick-Baez (KB) mirror assemblies. The large KB assembly features 320-mm long mirrors, which allows acceptance of a large incident beam—typically 500x500 μm^2 at 30keV — and focuses it down to approximately 5x5 μm^2 (FWHM). The small KB assembly can achieve a focused spot size of approximately 1x2 μm^2, thus offering superior spatial resolution (needed for samples at ultrahigh pressure or heterogeneous samples) at the cost of photon flux. Pinhole apertures ranging in size from ~10-100 μm in diameter are used to 'clean up' the focused beam, minimizing unwanted scattering from gasket or other surrounding materials.

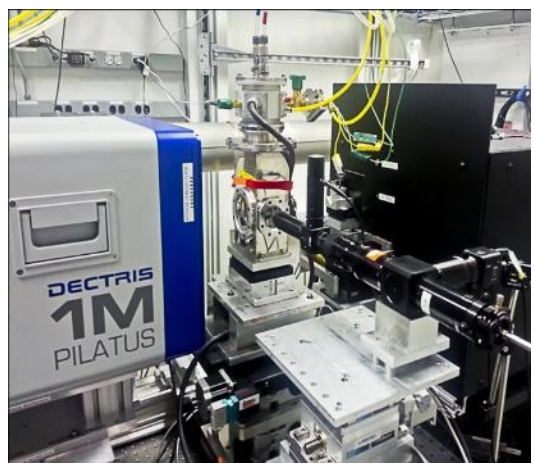

Fig. A1. Typical experimental setup at 16-ID-B with 320 mm KB mirrors, sample in resistevely heated DAC in vacuum shroud, online optical system, and Pilatus 1M detector.

The dimensions of DAC samples are typically on the order of microns or tens of microns while the mass of apparatus used for HP generation and sample environment control are typically on the order of kilograms or tens of kilograms. This contradiction of scales makes accurate and precise sample positioning quite challenging. The challenge is well met at 16-ID-B through the use of modular stage stack assemblies that can be quickly modified and optimized for the particular sample configuration and associated payload. For the majority of sample assemblies (up to 5 kg), high-speed, high-precision

motorized DC stages with submicron incremental encoders are used for rapid and repeatable sample positioning. For specialized applications requiring high-load sample assemblies (up to 50+ kg, for example large cryostats or high temperature furnaces), high load capacity stepper motor based stages are used with little sacrifice in speed and resolution. Recent developments include software and hardware implementation for high speed (on-the-fly) sample scanning with simultaneous x-ray transmission mapping and x-ray diffraction imaging. Near-future developments include the introduction of more complex, multi-axis sample trajectories during x-ray imaging. This greatly increases the number of crystallographic planes that contribute to (and thus, the structural information that can be extracted from) the x-ray image.

16-ID-B benefits from a diverse collection of large-format imaging detectors which offer great flexibility in terms of size, spatial resolution, dynamic range, and imaging frequency. The mar345 imaging plate is one of the largest area detectors available and it offers high resolution, low readout noise, and high dynamic range. The marCCD detector offers the smallest pixel size and relatively fast readout times for efficient data collection. The Perkin-Elmer amorphous silicon detector combines the largest detection area with imaging frequency as high as 15 Hz. The Pilatus 1M, HPCAT's flagship area detector, offers exceptionally high dynamic range, zero readout noise, and high frequency imaging at 125 Hz. Recent developments include sub-millisecond synchronization of the x-ray photon shutter with image collection, yielding the most accurate reflection intensities for single-crystal x-ray diffraction, and x-ray imaging coordinated with sample translation for rapid and comprehensive x-ray mapping of the entire HP sample chamber. Short-term developments (~1 year) hopefully include the acquisition of a next-generation CdTe-based photon counting detector similar to the Pilatus 1M, only with much greater detection efficiency and imaging frequency. Medium-term developments (1-3 years) include development of Soller slits system for studying structure of light elements, especially liquids and melts.

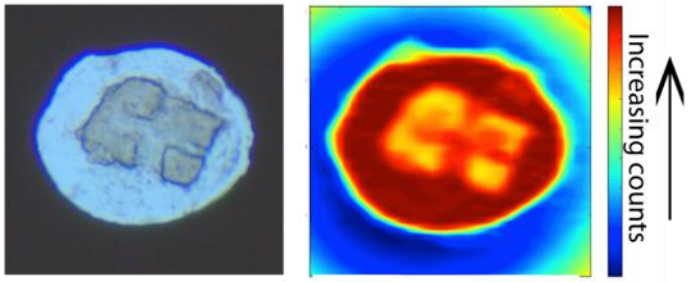

Fig. A2. Photo-micrograph (left) and 2D x-ray transmission map (right) of a multi-chamber diamond anvil cell sample.

The unique and varied set of experimental capabilities for structural studies of materials under extreme conditions (up to millions of atmospheres of pressure and from 4 to several thousands K) makes 16-ID-B a world-leading facility for addressing some of the most challenging questions in HP crystallography. For example, high load stages and high photon flux made cryogenic studies of lithium, one of the lightest elements, possible (Schaeffer et al., Nature Communications, 2015). The exceptionally high spatial resolution enabled the discovery of a new HP phase of nitrogen, synthesized at pressures greater than 100 GPa (Tomasino et al., Physical Review Letters, 2014). High frequency imaging combined with rapid, remote sample pressure control made it possible to explore the strain-rate-dependent kinetics of phase transitions in potassium chloride (Lin et al., Journal of Applied Physics, 2016). HPCAT is continually expanding the limits of possibility in the field of HP DAC crystallography.

[back]

6.1.2 HP x-ray diffraction – bending magnet beamlines [back]

The HPCAT 16-BM-D beamline is dedicated to high-pressure studies of physics and chemistry of matters using powder x-ray diffraction (XRD), single crystal X-ray diffraction (SXD), x-ray absorption spectroscopy (XAS), and anomalous X-ray scattering (AXS) techniques. The studies cover a variety of subjects including solid-state equation of state, phase transition, materials synthesis, materials characterization, high-pressure transport phenomena, and crystallography. The beamline is designed to accommodate various DACs as pressure generating devices so that the beam characteristics and sample/detector configuration are optimized to meet the constraints of the DAC environment.

For the XRD and SXD experiments, monochromatic X-rays are selected by a Si (111) double-crystal monochromator operated in an artificial channel-cut mode and focused to 5 µm × 5 µm (FWHM) by table-top Kirkpatrick-Baez type mirrors located near the sample stage. The range of X-ray energies covers 7-45 keV, with the maximum energy limited by the KB mirror cut-off angles. The maximum intensity at the sample is found at around 20 keV with ~10^9 photons/sec. The typical x-ray energy is around 30 keV with ~5×10^8 photons/sec. The instrumental resolution, $\Delta q/q_{max}$, reaches to 2×10^{-3}, and is tunable through adjustments of the detector distance and X-ray energy. The typical detector distance is around 320 mm but it can be flexibly adjustable depending on the experiment-specific requirement.

The XRD setup is compatible with high pressure sample environments ranging from ~4K cryogenic cooling to 1,000K resistive heating, as well as room temperature compression. The powerful versatility comes from a combination of high-quality optical components and reproducible alignment (on the order of a few microns) and is essential for optimizing each individual experiment. Despite the frequent changes required to meet users' requests, the beamline is operated in a fully optimized status. On-line microscope and Ruby system for in-situ pressure gauge, membrane or gear box for remote pressure control, and in-and-out ion chamber mount for X-ray absorption measurement are readily available.

The unique capability of 16-BM-D is a combination of simultaneous micro-XRD with high-energy XANES measurements, as well as the anomalous x-ray scattering / diffraction, which allows simultaneous probe of atomic and electronic structure at given sample conditions. The user-friendly interface and routine procedures developed for user operation allow easy switch between two different experimental setups. There is always an intimate relationship between the atomic and electronic structures; therefore measuring both at identical pressure and temperature conditions provides better insight into the high-pressure properties of matter.

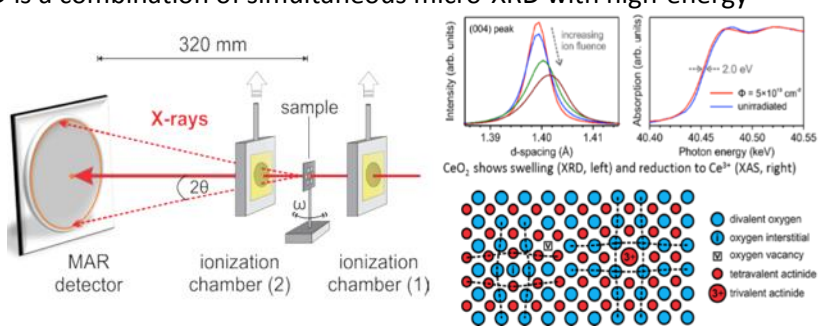

Fig. A3. (Left) Schematic illustration combined XRD-XANES with switchable setup; (right) correlated behavior between lattice distortion and redox state of cationic species, and illustration of Frenkel-type defect induced by ionizing radiation.

Recently the beamline was upgraded with a new 320 mm long KB focusing mirror; the MAR 345 detector was upgraded to decrease readout time and increase beamline efficiency. Currently the setup is being optimized with a new cleanup pinhole holder, combined PIN diode – beamstop assembly, and fly-scan capabilities to allow simultaneous fast diffraction/absorption mapping capabilities. The near term development plans include a new gas mixing setup for ion chambers to optimize the absorption measurements, redesign of the detector positioning cart to allow smaller sample-to-detector distances

and larger Q range, and installation of a beam positioning monitor in 16-BM-C to monitor and stabilize the X-ray beam position and allow more robust operation. Because of the photon flux limitation in BM beamline, the high-priority medium-term development plan includes possible replacement of Si monochromator with Germanium, which would double the flux on the sample due to ΔE/E bandwidth enlargement without compromising the Δq/q resolution.

The priority directions for 16-BM-D are single crystal (multigrain) diffraction and XRD-XANES combination, and this is reflected in multiple advanced studies and high-profile publications. Using the powder XRD technique integrating the resistive heating capabilities, Seoung et al. (Nature Chemistry, 2014) were able to suggest a possible chemical process in a geological setting that allows irreversible insertion of xenon into small pore zeolite, which may answer the long-lasting "missing xenon" problem. Extending the single crystal X-ray diffraction techniques to multi-grain diffraction patterns, the redundancy in intensity sampling that has been critically required for robust SXD analysis was achieved in an un-precedent level, which resulted in detailed electron density map for a high-pressure phase (β-Sn) of one of the most famous single element materials, Ge (R. Li, in preparation). Combining powder XRD and X-ray absorption spectroscopy measurements simultaneously at a given sample condition, Tracy et al. (Nature Comm., 2015) revealed the strain-related charge transfer mechanism in lanthanide metal oxide compounds.

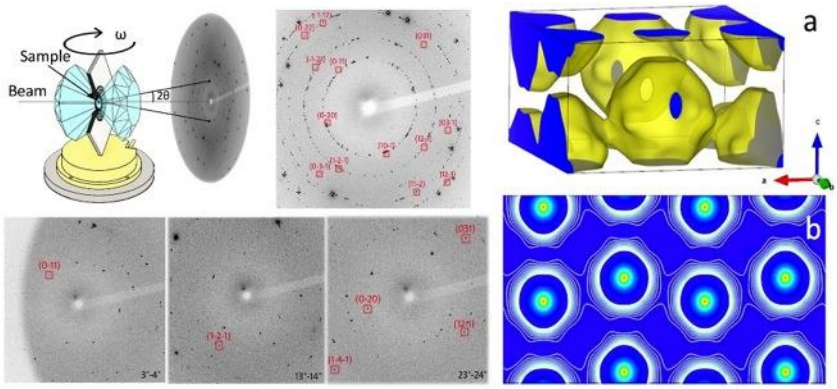

Fig. A4. (Left) Multigrain single crystal X-ray diffraction measurements – schematics and diffraction images; (right) 3D and 2D electron density distribution map of the β-Sn Ge.

[back]

6.1.3 HP in situ x-ray diffraction with laser heated diamond anvil cell [back]

The downstream end-station in 16-ID-B is an integrated system dedicated to micro-XRD combined with on-line laser heating (LH) techniques [270]. Laser-heated diamond anvil cell (LHDAC) coupled with the in-situ synchrotron x-ray diffraction is a unique and powerful experimental technique for studying a broad range of material properties (melting, phase transitions, thermal EOS) under extreme conditions up to megabars of pressure and several thousand degrees Kelvin of temperature. Over the last decade, this technique has evolved into a routinely used and productive experimental method at synchrotron beamlines, leading to numerous major scientific advances and a large expansion of high-pressure research in physics, chemistry, geoscience, and materials science.

Since this technique at HPCAT opened for user research more than 12 years ago, HPCAT staff have been continuously developing new LH techniques and conducting system upgrade and innovation. The established cutting edge and unique technical capabilities include the following specifications:

- Angle-dispersive micro-focused x-ray diffraction with energy range: 18 to 60 KeV, beam focus: 5 x 3 μm^2 FWHM, photon flux at sample: ~$3x10^{11}$ ph/s at 30 KeV, ~ 10^{12} ph/s at 20 KeV with 320 mm KB mirrors.
- Double-sided heating with two 100W infrared Ytterbium fiber lasers – continuous wave and power modulation.
- Separate temperature measurement from both sides with an imaging spectrograph and detectors of PIXIS 400BR CCD or PI-MAX4 (emICCD) for fast temperature measurement on microsecond time scale.
- In-situ variation of heating spot size (flat top area 4 to > 60 μm in diameter) to minimize lateral temperature gradient in heated sample.
- Mirror pinhole setup to allow directly observation of T measurement location relative to heating spot and XR beam to ensure the exact alignment.
- Modulation pulse heating synchronized with XRD, T measurements and thermal imaging.
- Remote pressure control, user-friendly control interface, and automatic data recording.

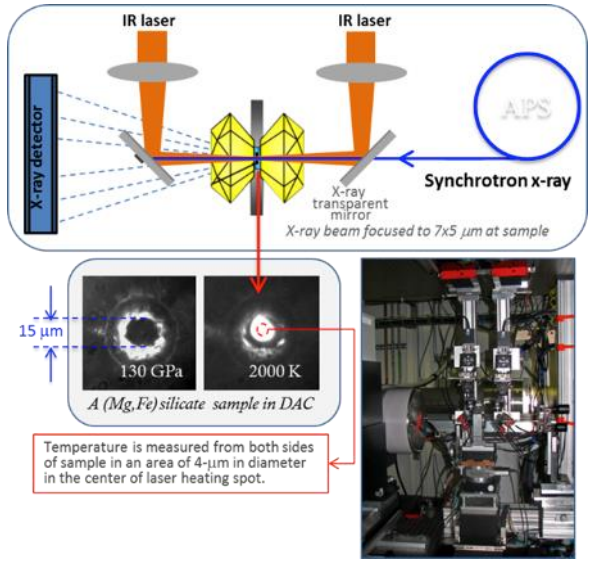

Fig. A5. Schematic diagram and photograph of a double-sided laser heating system in station 16-ID-B.

The technical development objective in recent years has been to advance the experimental capabilities that address specific issues in the most challenging areas of high-pressure research, specifically high-pressure melting, P-V-T EOS, and time-resolved studies. New techniques established at the beamline LH end-station of 16-ID-B in recent years include (1) on-line heating-spot size adjustment to provide effective and uniform heating on various-sized samples in the diamond anvil cell and minimize the effect of temperature gradient in phase transition and chemical reaction studies; (2) mirror pinhole setup to allow direct viewing of the temperature sampling area relative to the heating area and x-ray beam, and online adjustment to ensure the ideal alignment for reliable experimental measurements; and (3) modulated laser heating technique synchronized with XRD, fast temperature measurement and thermal imaging for high pressure melting and time-resolved studies of phase transition dynamics under high PT conditions; the short heating pulse together with the fast data collection also allow the diffusive scattering of melt to be captured before it disperses into surrounding matrix; it also minimizes potential chemical reaction caused by extensive CW laser heating. The short-term development projects include commissioning of fast (0.1-1 ms) temperature measurements system. The medium-term development projects include acquisition and installation of a more powerful pulsed laser (with sub-ms pulse duration) for higher temperature experiments at multi-megabar pressures and development of integrated pulse-probe LH μ-XRD techniques.

The unique set of experimental capabilities for structural studies of materials under extreme conditions (such as a combination of advanced laser heating and multigrain single crystal diffraction capability) makes 16-ID-B a world-leading facility for addressing some of the most challenging questions in HP crystallography. LHDAC integrated with in-situ synchrotron x-ray diffraction has been a highly demanded experiment technique and one of the most productive research areas at HPCAT. The development of this technique has resulted in a significant number of important scientific discoveries. For examples, it was shown that $(Mg,Fe)SiO_3$ perovskite is unstable under lower mantle conditions and transforms to $MgSiO_3$ perovskite and a new iron-rich hexagonal phase (H-phase), suggesting that

$(Mg,Fe)SiO_3$ Pv may not be the major silicate throughout the lower mantle and all geodynamic models will need to be revised (Zhang et al. Science, 2014). Another exciting study demonstrated that high pressure enables new chemistry and resulted in the discovery of previously unknown iron oxide compounds (Fe_4O_5, Fe_5O_6) (Lavina et al. Science Advances, June 2015). Use of newly developed multigrain single-crystal X-ray diffraction analysis technique in a laser-heated DAC at megabar pressures allowed the in situ single-crystal structure determination of $(Mg,Fe)SiO_3$ postperovskite (ppv) from a polycrystalline sample containing sub-micron ppv grains (Zhang et al. PNAS 2013).

6.1.4 HP x-ray Laue diffraction [back]

Experimental setup for polychromatic ("white beam") X-ray diffraction measurements at high pressures is available at APS 16-BM-B beamline (Fig. A6) [283]. To the best of our knowledge currently this is the only experimental facility around the world specifically dedicated for Laue diffraction mesurements at HP using synchrotron radiation. The major advantage of polychromatic beam diffraction compared to monochromatic is that comparable number of single-crystal reflections can be measured at least one-two orders of magnitude faster because there is no need to rotate the sample. This makes Laue diffraction a powerfull tool for real time studies of pressure induced microstructural changes, and time resolved studies of crystal morphology, texture and strain.

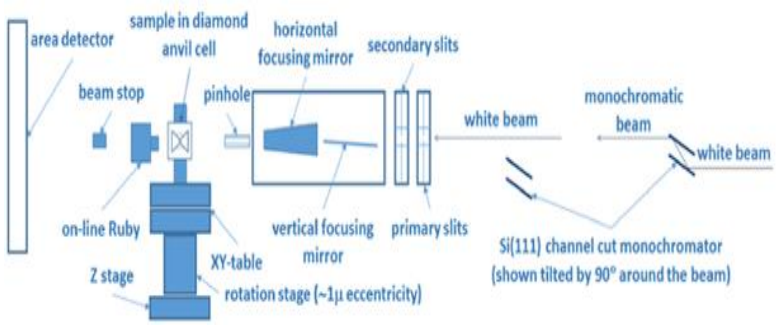

Fig. A6. Schematic of polychromatic X-ray diffraction setup at APS 16-BM-B beamline.

Reaching high Q is crucial for high-quality experimental results, in particular for accurate refinement of deviatoric strain. The typical x-ray energy range for white beam at 16-BM-B is 5-70 keV with the maximum energy limited by the KB mirror cut-off angles. This highest energy is significantly higher than at other Laue diffraction beamlines which makes it possible to perform Laue measurements in transmitted geometry as opposed to typical 90° geometry. This is beneficial for HP experiments because of DACs' and sample environment devices' angular constraints. HPCAT polychromatic beam setup also allows Laue measurements with 90° geometry in DAC providing that X-ray transparent gaskets or DACs with large opening are used. However Re gaskets used in transmitted geometry provide substantially higher pressure and finer pressure control than most transparent gaskets (e.g. Be). Also sample environment devises, such as cryostasts, currently can't be easily used in 90° diffraction geometry within an existing HPCAT Laue setup (this problem can be resolved given the adequate recourses).

For Laue micro-diffraction the x-ray beam is focused down to 5-10mμ^2 spot using a pair of 200mm Kirkpatrick-Baez (KB) x-ray mirrors. Diffraction patterns are usually collected with Perkin Elmer area

detector which is efficient for X-ray energies higher than 20 keV and has readout time in the order of 33-133ms. A compact Si(111) channel cut monochromator provides a complementary monochromatic beam which can be exchanged with the polychromatic beam at the sample position in a few minutes. The monochromatic X-ray beam is used for geometry calibration, to measure d-values of selected reflections, to identify powdered crystalline phases and to make indexation of Laue diffraction spots easier. The typical wavelength of monochromatic x-rays is 0.3065Å (40.45 keV).

High-quality spatially and temporally resolved single-crystal Laue diffraction for e.g. understanding mechanisms of phase transitions, requires accurate and reproducible sample positioning. The current reproducibility of the translational stages and eccentricity of the rotational stage are less than 0.5 µm and 1 µm, respectively. Sample pressure can be increased remotely with infinitesimally small steps with either a gearbox or membrane system and measured using on-line ruby fluorescence system. In order to get spatially resolved structural information and trace the structural evolution with time, a series of identical two dimensional (2D) grid scans can be made after each pressure change. In each 2D grid scan, Laue diffraction images are collected at steps comparable to the X-ray beam size and with typical exposure time of 0.5 s. Collection of one 2D translational scan covering a DAC sized sample usually takes a few minutes.

HPCAT polychromatic beam setup was recently implemented to study mechanism of pressure induced α→β phase transition in Si to demonstrate characterization of morphology, relative orientation and deviatoric strain of crystals in real time mode [283]. Angles between crystals can be measured with precision down to ~0.02° from positions of individual diffraction spots. With the currently available ratio of pixel size over sample to detector distance, the precision of the relative crystal orientation can be one order better and the precision of deviatoric strain tensor components can be better than 3×10^{-4} if perform refinement of these parameters against positions of all the available diffraction spots. Time resolution ranges from seconds to minutes depending on the size of the studied area. In the mentioned example of α→β phase transition in Si notable microstructural changes have been observed in minutes, while collection of one translational scan of the same size and resolution as in this case study using monochromatic beam would take hours to days, because angles between crystals were smaller than 0.21° and measuring of such small angles with monochromatic beam would require very fine slicing.

Currently we are building a completely new dedicated HP Laue setup in 16-BM-B which will be commissioned in September 2016. In addition to improved stability, flexibility in detector positioning, and general use-friendliness, the new setup will be based on KB mirrors focusing X-ray beam down to 1×2 $m\mu^2$. This will drastically improve spatial resolution bringing it to the level of typical size of dislocation cells. Due to this samples which produce streaky reflections with the current setup will produce substantially sharper diffraction spots making their analysis much easier and providing significantly more information. The medium-term development project include development of new DACs and sample environment devices allowing high-quality Laue diffraction at elevated and cryogenic temperatures.

Multiple case studies are currently being conducted at HPCAT in order to promote high pressure Laue diffraction and to optimize data analysis procedures for routine beamline operation. For example unusual phenomenon was observed in ultrapure Zr: the sample which was originally fine powder becomes much grainier after α→ω phase transition. In order to understand nature of this phenomenon some real time Laue data across the phase transition were collected and the data currently are being analyzed (collaboration with Nenad Velisavljevic, Los Alamos National Laboratory). Mechanisms of pressure induced phase transitions in the following materials are being studied: Fe, Si, forsterite

(collaboration with Joel Bernier, Lawrence Livermore National Laboratory), Ferrosilite (collaboration with Przemek Dera Hawai'i Institute of Geophysics and Planetology).

6.1.5 HP inelastic x-ray scattering [back]

The spectroscopy beamline at 16-ID-D utilizes a spectrometer with backscattering analyzers to explore the electronic structure and excitations of systems under high pressure.

One of the established experimental technique is X-ray Raman. Photons in the hard x-ray regime impinge on the sample and transfer energy and momentum to a core electron, exciting it to an unoccupied state. This non-resonant technique is element specific, probes bulk systems and because of the use of hard x-rays of about 10 keV, it can investigate the core-excited states of systems under high pressure in diamond anvil cells. At small photon momentum transfer for light elements, the photon energy-loss spectrum approximates the x-ray absorption spectrum. The measurement of the dynamic structure factor yields the near-edge absorption fine structure when the photon energy loss is near the binding energy of the core electron under study.

At the spectroscopy station 16-IDD, we employ a two different X-ray Raman experimental configurations. In our standard setup for relatively high count-rate measurements, we use a 17-element backscattering analyzer array, post-sample collimating slits to reduce parasitic scattering from the diamond anvil cell gasket, and a single element Silicon detector. Each analyzer in the array is a 2-inch diameter Si(111) wafer bent to 1-meter diameter, aligned on the Rowland circle. The second configuration uses a post-sample focusing polycapillary to collect a large solid angle of scattering, after which the scattered photons are analyzed by a single 4-inch diameter backscattering analyzer. Fig. A7 below shows some of the results measured on our beamline.

We are developing a spectrometer to explore single-particle and collective electronic excitations of the electron gas. Equilibrium properties are a starting point for the understanding of materials. But, the electronic excitation spectrum of materials is crucial to explaining their behavior and dynamical properties. Measuring the photon energy-loss in the (sub-) eV resolution range as a function of

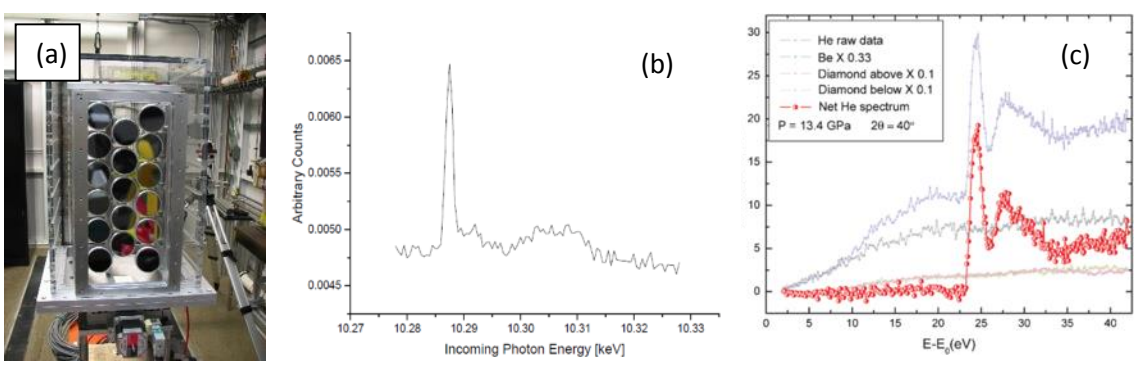

Fig. A7. a) the 17-element backscattering analyzer array, b) K-edge X-ray Raman from N_2 at 80.3 GPa [L. Wang, unpublished], c) Helium exciton.

scattering direction, S(**q**,ω), yields important information about the electron gas with relatively

straightforward data analysis (compared to resonant IXS). Via a Kramers-Kronig analysis, the dielectric function ε(**q**,ω) can be extracted. Fundamental properties can be investigated, such as valence excitations, single particle (inter- and intra-band) transitions, quasi-particle excitations, plasmon (collective) excitations, energy-momentum dispersion relations of excitations, screening, exchange and correlation effects. A measurement of S(**q**,ω) for low-q, under extreme conditions is for the most part un-explored yet. Our aim is to build a non-resonant Inelastic X-ray Energy-Loss Spectrometer optimized for the study of the medium energy loss (~0-30 eV), medium energy resolution (50-100 meV), low momentum transfer ($q < q_c$, about 2.5/Ang), for the study of Elementary Electronic Excitations of the Electron Gas in Bulk Systems at High Pressure and low temperature.

We measure the energy loss of the x-ray photon as a function of scattering angle. The incoming x-ray photons exchange energy and momentum with the electron system (inelastic scattering). The spectrometer is aligned to measure photons of a fixed energy, within narrow energy resolution width. We scan the upstream monochromator(s). The difference between the incoming photon energy and the fixed analyzed energy is the energy loss of the photon, which is the excitation energy gain of the electron system. Using a post-sample polycapillary, scattered photons within the polycapillary's input solid angle are collected, channeled and focused, then continue to a backscattering energy analyzer. The polycapillary preserves the scattering angles, so the energy analyzed photons end up in the Pilatus pixel area detector with known scattering angle. In this way, we can simultaneously map significant regions of q-space as a function of energy loss.

A sketch of the working concept of the spectrometer is shown on Fig. A8, along with preliminary results. In the upcoming run starting in June, we will be determining the instrumental corrections, necessary for a quantitative determination of the dynamic structure factor. Then we will develop the software to analyze the image data, multiplex the polycapillaries, and proceed to higher energy resolution.

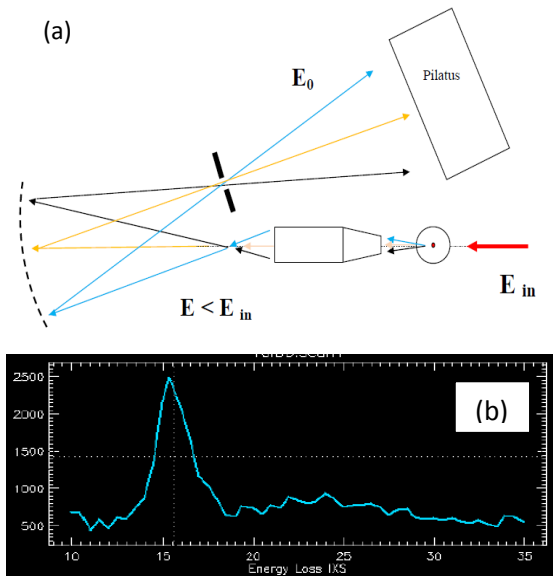

Fig. A8. a) schematic of the backscattering spectrometer using a post-sample polycapillary, backscattering analyzer and area detector. b) preliminary results—bulk plasmon of Al at 2 GPa.

[back]

6.1.6 HP x-ray emission spectroscopy [back]

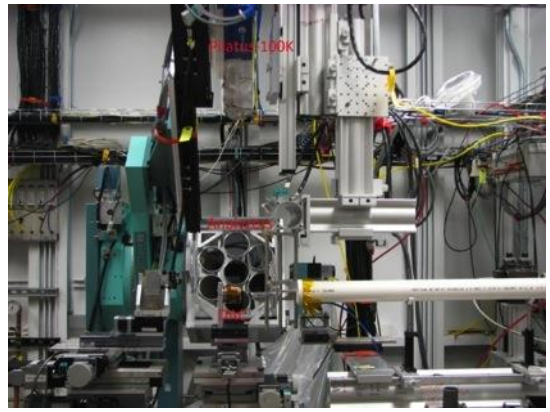

Fig. A9. Photography of 7-element XES spectrometer at 16 ID-D station.

X-ray emission spectroscopy (XES) is a photon-in-photon-out process. During the process, a core electron is excited by the incident X-ray and the core hole is filled by an electron from a higher shell. Another photon, whose energy equals the energy difference between the two electronic levels, is emitted. Different from X-ray absorption spectroscopy (XAS) that probes the unoccupied states, XES probes the occupied electron levels and can provide valence, spin and bond information of the system studied. If the emission spectrum is measured with an incident X-ray energy close to the absorption edge of the studied material, there is a narrowing effect due to the resonant excitation; it is called resonant X-ray emission spectroscopy (RXES). Partial fluorescence yield X-ray absorption (PFY-XAS) spectrum is collected at fixed emission energy while scanning the incident X-ray energy across the absorption edge. Similar to RXES, PFY-XAS has a sharpening effect due to reduce in core-hole life time broadening. RXES and PFY-XAS studies of transition-metal and *f*-electron systems give us important information on the electronic states of the systems, such as the valence, band-width and crystal fielding splitting.

In our current XES setup (Fig. A9), an emission spectrum is collected by a 1-meter Rowland circle XES spectrometer in the vertical scattering geometry. In Aug 2015, we just upgraded our XES spectrometer which contains up to seven 4-inch Si analyzers and a Pilatus 100k position sensitive detector. A range of emission lines (4.5-21keV) can be studied by selecting Si analyzer with right index. Since 2008, we have built a cryostat which can reach down to ~10K for R/XES studies on magnetic properties and spin transitions of many functional materials such as superconductors under high pressures and low temperatures.

Since its first application under high pressure in the late 1990s, XES has been widely used to study the behavior of materials under high pressure. At HPCAT, HP XES has found applications in many high pressure scientific fields and has been used to probe spin transition of mantle minerals (geophysics), local spin moment of superconductors (material sciences), valence band of semiconductor (condensed matter physics) and oxidation state of *f*-electron systems (chemistry). As an example, the comparison of RXES data to theoretical fits at different pressure for Kondo insulator SmB_6 show that the *f* occupancy was reduced with increasing pressure; however, Sm maintains a significant divalent character up the highest pressure studied 35GPa. Thus, the closure of the resistive activation energy gap and onset of magnetic order are not driven by stabilization of an integer valent state.

Although our current XES spectrometer has 7 analyzers, only 6 emission lines (one emission line for each Si index) can fully use all 7 analyzers because we don't have motorized angles and radial movement for each analyzer. It took 1-2 days to align all 7 analyzers for an emission line and it is not practical for general user operation to use all 7 analyzers for every emission line with current design. We are in the process to build a new spectrometer with motorized analyzer angles and radial translation. Our goal is to align all 7 analyzers in 3-4 hours with automatic scan function.

High pressure XES will greatly benefit from the developments in new synchrotron radiation sources and better X-ray focus optics. The new diffraction-limited storage rings has ultra-low emittance in both

horizontal and vertical directions (~50pm-rad for APS-U) and can provide a much smaller and intense beam down to sub-micrometers. Recently, the superconducting transition-edge sensor (TES) with high energy resolution has been developed. A single TES can reach an energy resolution of 1.6eV FWHM at 6keV which is comparable to that of a typical XES spectrometer using crystal analyzer. Using such TES arrays for XES measurement will no longer require the use of crystal analyzers, dramatically simplifying the experimental setup. All these future developments will dramatically expand the XES capabilities for high pressure research, including position scanning imaging, time resolved measurements, and multi-megabar measurements.

6.1.7 HP nuclear resonant scattering [back]

The nuclear resonant scattering (NRS) technique has been widely used to study biomolecules, thin films and materials under extreme conditions since middle 1990s. NRS can be divided into two methods: nuclear resonant inelastic X-ray scattering (NRIXS) and nuclear forward scattering (NFS). NRIXS is a technique in which energy gain or loss through a scattering process involving phonon excitations is recorded via excitation of a Mössbauer resonance. Apart from the familiar 'zero phonon' Mössbauer resonance which NFS is based on, there are additional transitions that correspond to nuclear excitation in combination with excitation (Stokes) or de-excitation (anti-Stokes) of phonon modes. From NRIXS, the partial density of states (PDOS) can be obtained to derive important dynamic, thermodynamic, and elastic information such as vibrational kinetic energy, force constant, Debye temperature, and sound velocities. From NFS, hyperfine interaction parameters can be obtained to give information on spin state, oxidation state and magnetic ordering.

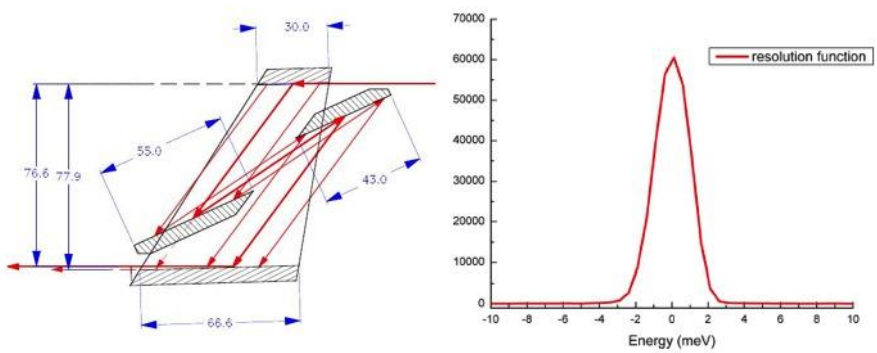

Fig. A10. (a) Schematic drawing for 2meV HRM for ^{57}Fe nuclear resonance at HPCAT; (b) resolution function of the HRM.

At HPCAT, the bandwidth of the undulator beam is reduced to ~1eV by a liquid N_2 cooled Si (1 1 1) double crystal monochromator (DCM). After going through the high resolution monochromator (HRM), the bandwidth of X-ray beam is further reduced to ~2.2-meV. The HRM for ^{57}Fe nuclear resonance at 14.413keV consists of 2 channel-cut silicon crystals [Si(4 4 0) and Si (9 7 5)] (Fig. A10). In addition to the pressure dependent studies at room temperature, we have two cryostats coupled with in-situ pressure control and online ruby system to do NFS experiments at low temperatures and high pressures. In Dec 2015, we have installed and commissioned a new set of KB mirrors to get focus size of 3(V) x 5(H) µm FWHM. With such small focus, NFS mapping experiment becomes feasible.

The NRS technique is selective for particular isotopes. Only samples with resonant nuclei contribute to the signal. This isotope selectivity is very helpful for high pressure studies by suppressing the background signals, where samples are normally enclosed by surrounding materials (gasket, diamond,

etc.). At 16 ID-D of HPCAT, we have applied NRS to many applications (studying lattice dynamics, valence and spin state of perovskite and post-perovskite, magnetic transition of Fe-based superconductors, isotope fractionation of Fe metal and its alloys etc.). As an example, temperature-dependent NFS spectra of $BaFe_2As_2$ in Ne pressure medium at 5.3GPa show quantum beats in the spectra below 110K represent the occurrence of the magnetic fields and hence the antiferromagnetic state, whereas the absence of beats above 110K indicates the paramagnetic state. By combining with XRD measurements, these results show that magnetic ordering precedes the structural transition at high pressures and can be understood in terms of spin fluctuations in the emerging nematic phase before long-range magnetic order.

Nuclear resonant scattering experiments require not only the high brilliance of a synchrotron and a meV bandwidth HRM, but also a suitable time structure of the synchrotron. To distinguish the NRS signal from other much stronger electronic scattering, fast time discrimination electronics and an APD (~ns temporal resolution) are used. APS 24-bunch mode which has a 153 ns time separation between two synchrotron pulses is used for all NRS experiments done at HPCAT currently. From the initial design parameters of the new APS-U multi-bend achromatic (MBA) ring, the largest time separation will be ~76ns which does not significantly impact NRS experiments of isotopes with a few tens of ns half-life like Eu, Sn and Sm. For the most commonly measured NRS isotope ^{57}Fe which has 141ns half-life, this time separation is still good enough for NRIXS experiments which integrate signals from the entire time-collection window. However for NFS experiments, the 76ns time window is too short to collect useful time spectra. Measurements have been done at the APS with 1kHz high speed shutter to prove the principle. Without a high speed shutter, a HRM is needed to reduce bandwidth of the incoming beam to meV level in order not to saturate the forward APD detector with direct beam. Incident beam intensity is reduced ~500-1000 times by the HRM. With improving high-speed shutter technology and smaller focus beam, a factor of ~1000 improvement in collecting efficiency is possible.

6.1.8 Paris-Edinburgh press facility [back]

The Paris-Edinburgh (PE) press facility has been developed in 16-BM-B for comprehensive studies of liquids and amorphous materials at high pressures and high temperatures. Knowledge on the structure and physical properties of liquids and amorphous materials is important in various scientific fields such as condensed matter physics, material science, and geoscience, as well as in engineering and industry. Efforts have been made to investigate the structure and physical properties such as density, viscosity, and elastic wave velocity of liquids and amorphous materials. However, such experimental studies under high-pressure and high-temperature conditions remain technologically challenging.

An integrated setup for liquid structure, elastic wave velocity, and viscosity measurements has been developed using a PE press at the 16-BM-B station in HPCAT [254,255]. The PE press allows for compressing large volume samples (up to 2 mm in both diameter and length) up to ~7 GPa and 2000 °C, which enables us to investigate structure and physical properties of high-melting temperature materials such as metals and oxides. Multi-angle energy dispersive X-ray diffraction provides structure factors of liquids and amorphous solids to a large momentum transfer (Q) of ~20 $Å^{-1}$. Ultrasonic techniques have been developed to investigate elastic wave velocity combined with the X-ray imaging. Falling sphere viscometry, using high-speed X-ray imaging (up to ~105 frames/second (fps) in air and up to ~104 fps in

PE press), enables us to investigate a wide range of liquid viscosity, from those of high viscosity silicates or oxides melts to low viscosity (<1 mPa s) liquids and fluids such as liquid metals or salts. In addition to the falling sphere viscometry, white X-ray imaging technique allows for imaging phase separation and immiscibility of liquids at high pressures not only by density contrast but also by phase contrast imaging in particular for low density contrast liquids. In addition to these established techniques, the setup to measure density of liquids and amorphous solids at high pressures is under development. The integration of these multiple techniques has promoted comprehensive studies of structure and physical properties of liquids as well as amorphous materials at high pressures and high temperatures, making it possible to investigate correlations between structure and physical properties of amorphous solids and liquids. The PE press facility at the 16-BM-B station is unique beamline optimized for comprehensive study of liquids and amorphous materials at high pressures and high temperatures.

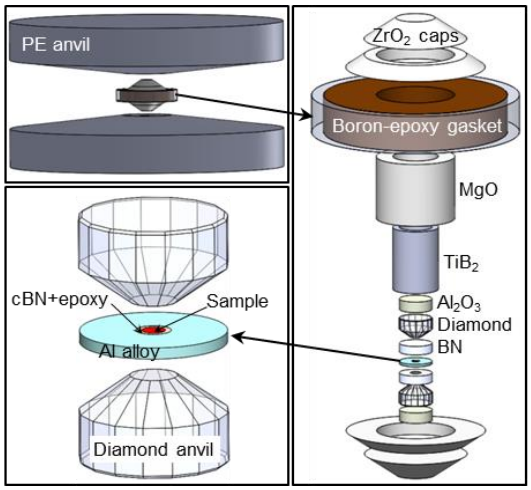

Fig.A11. Newly developed double-stage large volume cell.

In addition to the established PE press experiments, recent development of double-stage large volume cell (Fig. A11) enables us to compress large glass samples to near 100 GPa and to conduct multi-angle energy dispersive X-ray diffraction measurement for *in situ* structure measurements (Kono et al., PNAS, 2016). A research on structure of GeO_2 glass investigated using the newly developed technique revealed new experimental evidence of ultrahigh pressure polyamorphism in GeO_2 glass with coordination number of Ge significantly greater than 6 (Fig. A12). The new experimental data provides new insights into structural changes in network-forming oxide glasses and liquids at ultrahigh pressure conditions.

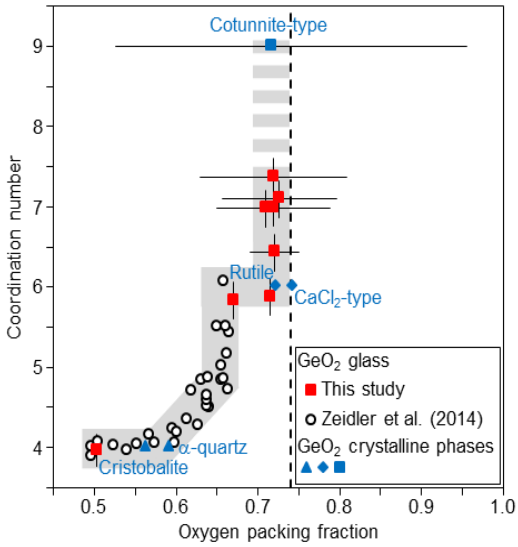

Fig. A12. Relationship between the oxygen packing fraction and coordination number of Ge in GeO_2 glass.

Further development of double-stage large volume cell is ongoing towards higher pressure generation and/or large sample volume compression. For example, use of 0.6 mm culet diamond anvil enabled to generate pressure higher than 100 GPa (up to 119 GPa) with large 0.2 mm diameter sample. The near-term development efforts are concentrated on further advancing the double-stage large volume cell technique in PE press with X-ray diffraction measurements on weak scattering materials such as low Z samples, glasses, and liquids. Medium-term development projects include design and installation of a focusing Laue monochromator to increased x-ray flux on the sample for more efficient and faster imaging capability and development of Soller slits setup for structure measurement on low-Z samples, especially liquid structure.

The PE-program in 16-BM-B has been extremely successful and very productive. This is manifested in a large number of publications, a significant number of which is published in high-profile journals. Notable works include determination of ultralow viscosity of carbonate melts at high pressures (Kono et

al., Nature Commun., 2014), measurements of density of metallic glasses and their pressure dependence (Zeng et al., PRL, 2014), observation of liquid-liquid phase transition in Cerium determined by equation of state measurements by radiography (Lipp at al., JPCS, 2014), and in situ x-ray diffraction, electrical resistivity and thermal measurements using a Paris-Edinburgh cell (Baker et al., JPCS, 2014) to name a few.

6.1.9 Laboratories and support equipment [back]

The success of high-pressure experiment mainly depends on the quality of the sample and high-pressure assembly. Typical Diamond Anvil Cell samples have dimensions of only several to several tens of microns with the total sample assembly, including pressure markers, insulation layers, etc) in the order of sub-millimeters. Thus sample preparation for successful experiment requires not only skillful hands, but a significant number of sophisticated pieces of equipment. HPCAT is a user facility with more than 230 individual users each year and more than 570 "person-visits" per year. Many users prepare their samples in their home institutions, but the majority of users use HPCAT sample preparation lab because they either do not have a comparably equipped facility in their home institution, or because of the complexity of the experiments which require multiple loading and reloading of the sample. Also samples and/or pressure devices may be subject to unpredicted changes or even failures as experimental conditions vary. Therefore it is a priority for HPCAT to maintain a state-of-the-art sample preparation facility.

Fig. A13. Laser drilling / micromachining system and Microsupport micromanipulator

HPCAT sample preparation laboratory is fully equipped for successful DAC and sample preparation and DAC loading in different sample environments. The major equipment include three Leica microscopes with variable objectives, glovebox with Argon atmosphere and a micromanipulator, state-of-the-art computer controlled Microsupport micromanipulator with a set of all essential tools, vacuum furnace and various desiccators, micro EDMs and mechanical drilling machine, ultrasonic cleaners, micro scale, other essential lab equipment. The laboratory has a special Beryllium handling area with a dedicated microscope and EDM machine. The highlight of the sample lab is a recently added Laser drilling / micromachining system[242] which was designed and built by HPCAT staff (Fig. A13). In cage area we have Rolan mini-CNC mill for PE cell sample assemblies preparation, grinding/polishing machine and other equipment for preparation of high-temperature DAC and other setups. This combination of the equipment with excellent housekeeping and exceptional user support makes the sample loading laboratory a unique and most sophisticated facility or a kind worldwide.

The advancement of HP science requires more complicated and sophisticated samples assemblies (i.e. multistage diamond cells, multilayer engineered sample assemblies, etc.) and therefore the sample preparation lab is constantly evolving to keep up with the sample strict loading demands. Near term plans include addition of small equipment items such as an electro-polisher for sharpening micromanipulator/sample loading needles and high-temperature vacuum furnace. Long-terms plans include purchase and installation of a new micromanipulator in the glovebox, and acquisition of

femtosecond laser and thermal evaporation/sputter deposition system for preparation of engineered samples and sample assemblies.

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.

Because the ability to remotely control pressure in DACs in accurate and consistent manner at various temperatures is crucial for effective and reliable operation of a high-pressure synchrotron facility such as HPCAT, over the last several years a considerable effort has been made to develop instrumentation for remote and automated pressure control in DACs during synchrotron experiments. HPCAT staff has designed and implemented an array of modular pneumatic (double-diaphragm), mechanical (gearboxes), and piezoelectric devices and their combinations for controlling pressure and compression / decompression rate at various temperature conditions – from 4 K in cryostats to several thousand Kelvin in laser-heated DACs. Such devices can be used for automated data collection along predefined P-T paths. The development effort has been focused on creating different loading mechanisms and frames for a variety of existing and commonly used diamond cells rather than designing specialized or dedicated diamond cells with various drives. We actively continue developing new mechanisms and setups for reliable and efficient pressure control suitable for different DACs and broad range of experimental conditions. The near-future projects include development of a DAC integrated with piezo-control for single-crystal diffraction measurements, and development of piezo-based dynamic DAC for cryogenic temperatures, and design of DACs with double-membrane pressurizing-depressurizing mechanisms with either 80 degrees symmetric axial openings and 70 degrees side openings for single-crystal and radial diffraction. The remote-future projects include the integration of pressure control with pressure-measurement devices and automation of pressure control process.

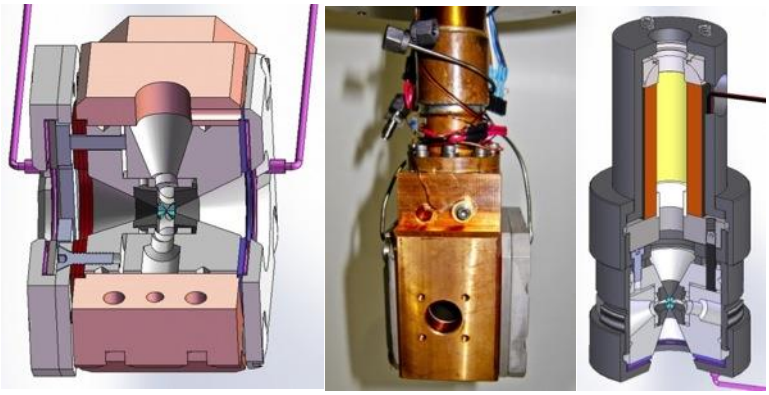

Fig. A14. (Left) Dual ("push-pull") double-diaphragm pressure control frame for compact cryostat; (right) Dynamic piezoelectric drive in decompression mode and double-diaphragm assembly in compression mode coupled with symmetric DAC.

A significant effort have been put into developing devices for temperature control in DAC, i.e. cryostats and heaters. In addition to existing cryogenic facilities, we have designed and implemented a variety of compact cryostats for different synchrotron techniques (powder and single crystal diffraction, inelastic scattering, etc.) The cryostats can accommodate a variety of standard and novel DACs, can be easily integrated with remote pressure control devices, and allow for high-pressure measurements at temperatures down to 2-4 K. The short-term development projects include constructions of a compact cryostat for Nuclear Resonant Inelastic X-ray Scattering for temperatures below 10K and with integrated membranes pressure control; and a new modular compact combination cryostat with stable sample position (zero drift during temperature change) and multi-function capability (e.g. it can accommodate various types of diamond cells with double-membrane setups for single crystal diffraction, high-efficiency Raman, and other experimental, mostly x-ray, techniques) and allow a variety

of novel types of unique experiments such as piezo-driven pressure control at cryogenic conditions which have not been possible before. The longer-term projects include construction of a new direct flow cryostat optimized for single crystal diffraction and ISX measurements down to 2K, and a new cryostat for IXS with polycarpellary X-ray optics.

In addition to cryostats, HPCAT has several whole-cell resistive heaters which can bring a DAC to 600-700K in air and up to 1000K in vacuum enclosure. A short term project includes design of a whole-DAC in-vacuum heating system compact based on compact sheathed coil heaters which allow to decrease heating and temperature stabilization time by up to an order of magnitude and decrease heater weight by several pounds. The long-term project includes development double-stage heating based on both multilayer engineered graphite and metal heaters.

For fast pressure measurements via ruby fluorescence and Raman shift of diamond, as well as for in-situ/ex-city Raman sample characterization during synchrotron experiments we have designed a number of portable online and stationary optical systems for such as ruby fluorescence pressure measurements and. Combined with remote pressure control systems, they significantly increase productivity of beamlines during high-pressure experiments. The pool of portable optical systems include four 12X zoom Navitar bases ruby systems and one compact Raman system. The near-term upgrade projects include building an upgraded version of a high-efficiency online Raman system with direct laser feed and upgrading all online ruby system and replacing low-efficiency single-mode optical fiber laser feed with a robust and efficient direct laser feed. The long-term projects include the development of upstream ruby/Raman system for simultaneous optical and x-ray measurements and portable laser heating / IR temperature measurement system. The pool of stationary/offline optical systems consists of a hallway ruby system, laser-heating system and Raman system. The near-future projects include overhaul of the stationary laser-heating system and turning it into a user-friendly Class I versatile instrument for sample synthesis and annealing. The long-term plans include reconstruction of the stationary Raman system to increase versatility, sensitivity and resolution by adding two new lasers (blue and red) and redesigning the optical layout.

Fig. A15. (a) HPCAT hallway offline ruby system and (b) portable online ruby system in 16-BM-D.

Even though the support equipment is routinely used in the majority of HPCAT experiments, there are multiple examples where the experiments would not be possible without specific equipment developed at HPCAT. For example, HPCAT-designed compact cryostat optimized for single crystal angle dispersive x-ray diffraction was used to map the phase diagram of elemental Lithium at HP low T (Schaeffer, Nature Comm., 2015). The development of fast compression-decompression devices for time-resolved measurements in DACs allowed studying the dependence of phase diagram on Ge and Si on decompression rate (e.g. Haberl et al., PRB, 2014), and kinetics of pressure-induced phase transition in KCl as a function of the compression/decompression rate (Lin et al., JAP, 2016). A combination of x-ray diffraction with online optical Raman spectroscopy has resulted in the new experimental direction of x-ray induced HP chemistry (e.g. Pravica et al., APL, 2013; Pravica et al., JCP, 2014).

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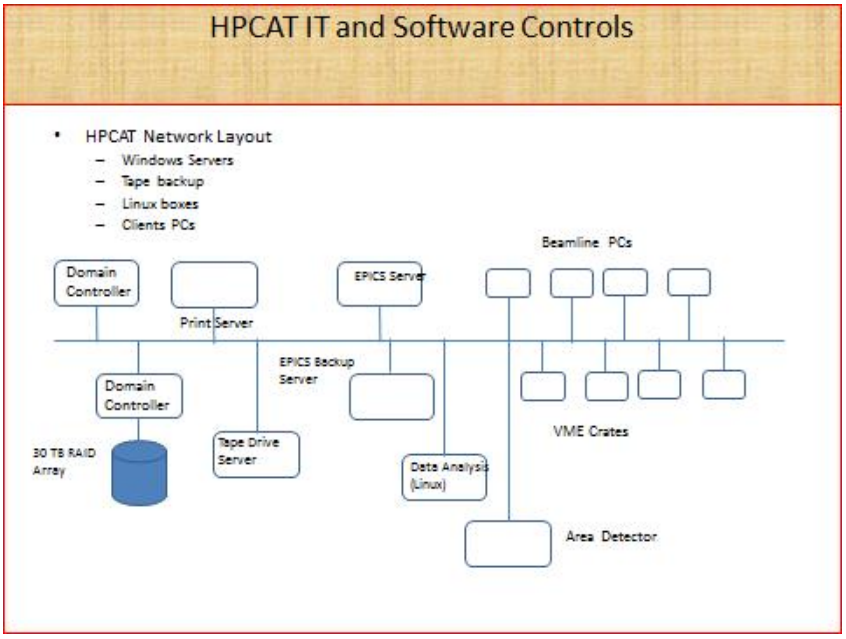
6.1.10 Beamline controls and data management [back]

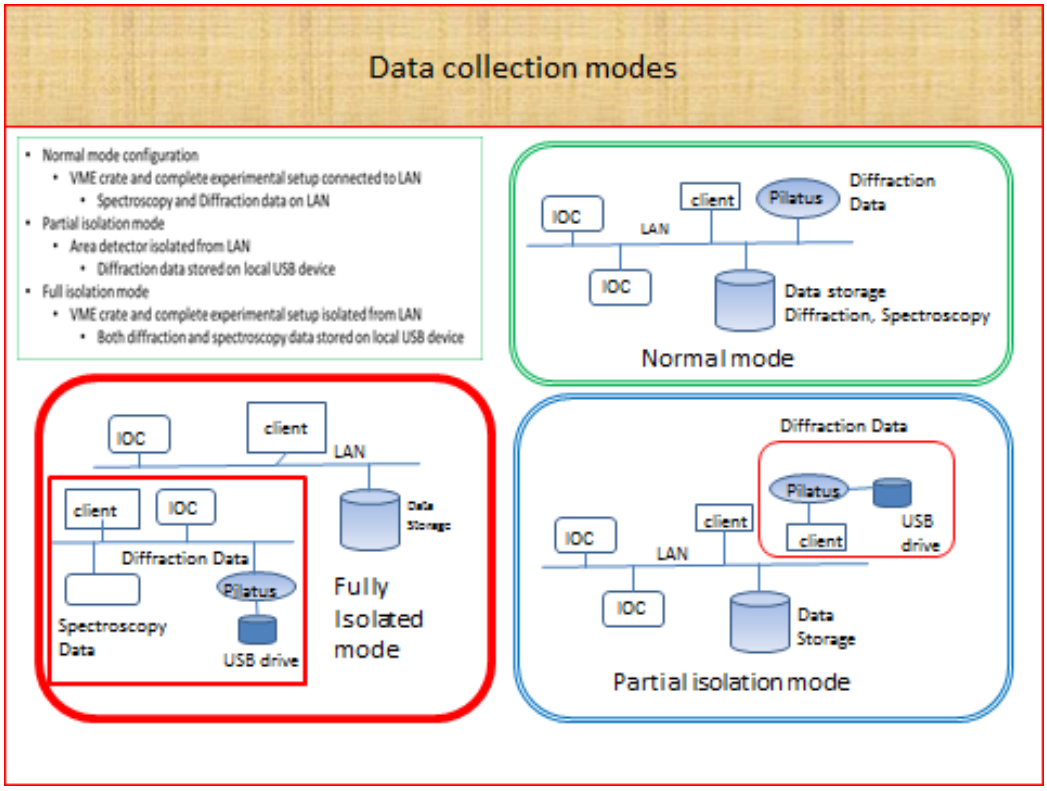
Computer support at HPCAT comprises of two pronged approach. First is the underlying domain infrastructure that provides an domain level network of servers and clients providing a small office environment and second, a distributed VME crates and associated software that control hardware devices for four beamline controls and data collection. Five Windows Servers and three Linux boxes form the backbone of HPCAT domain. There are over 30 windows client machines in use at office and at beamlines. We have a 30 TB storage RAID Array to store all the files (Office work and experimental data). Each night all files are backed up on to a Quantum SuperLoader3 LTO-6 tape library.



Data collection at HPCAT is built on EPICS (Experimental Physics and Industrial Control System). The client-server system (EPICS) is a proven architecture used in most synchrotrons around the world and at HPCAT we have added custom made software to assist in data collection and management. We have four fully and independently operational beamlines, each of which has a VME crate which houses various boards that communicate with devices that operate the beamline.

Apart from the default data collection mode, a partial and a complete network isolated modes are provided that allow secure data collection during experiment.

On site software: HP experiments are often dynamic; users need to make decisions based on the collected data so far. It is essential to have software for timely data evaluation on site. HPCAT has developed a number of software routines for determinations of pressure, temperature, unit cell parameter, etc.


Data collection modes
Normal mode configuration
VME crate and complete experimental setup connected to LAN
Spectroscopy and Diffraction data on LAN
Partial isolation mode
Area detector isolated from LAN
Diffraction data stored on local USB device
Full isolation mode
VME crate and complete experimental setup isolated from LAN
Both diffraction and spectroscopy data stored on local USB device
IOC
client
LAN
Pilatus
Diffraction Data
IOC
Data storage
Diffraction, Spectroscopy
Normal mode
IOC
client
LAN
client
IOC
Diffraction Data
Pilatus
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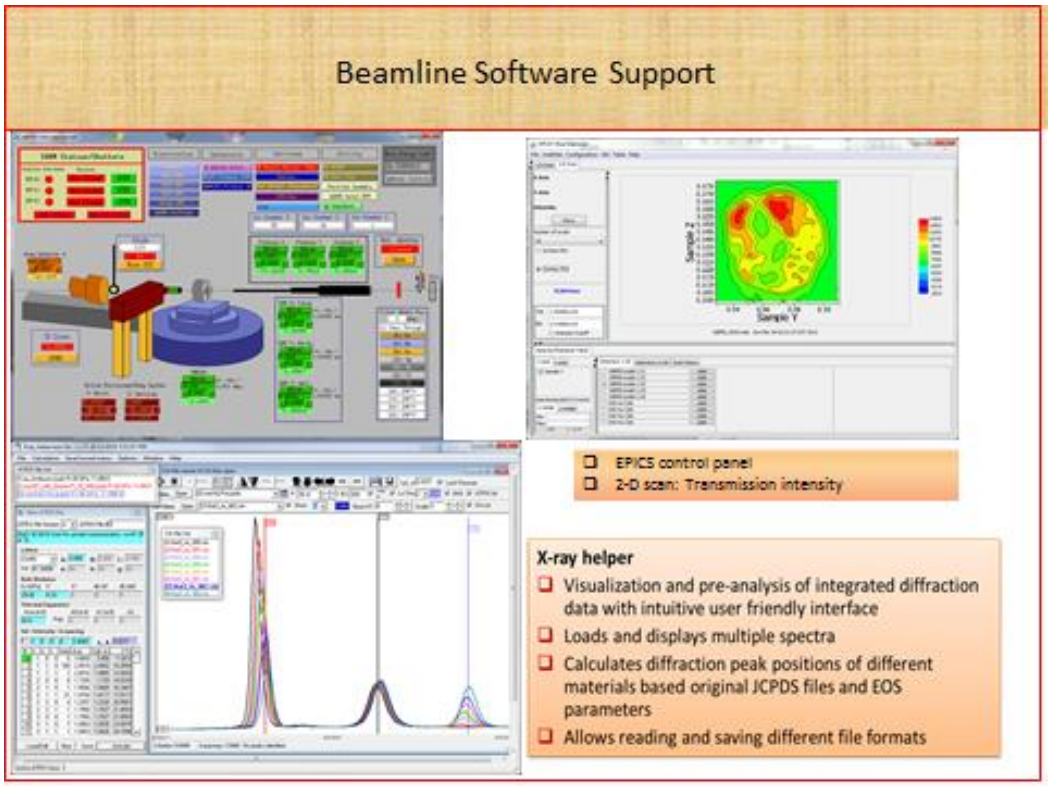
IOC
LAN
IOC
Data Storage
Partial isolation mode


Beamline Software Support
EPICS control panel
2-D scan: Transmission intensity
X-ray helper
Visualization and pre-analysis of integrated diffraction data with intuitive user friendly interface
Loads and displays multiple spectra
Calculates diffraction peak positions of different materials based original JCPDS files and EOS parameters
Allows reading and saving different file formats

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APPENDIX 6.2: 2015 Report of the HPCAT Advisory Committee

Report of the HPCAT Advisory Committee:

Date of meeting:	June 1, 2015
Location of meeting:	Advanced Photon Source, Bldg. 434, Rm. C010
Committee members:	Wolfgang Sturhahn (Chair), California Institute of Technology Michael Desjarlais, Sandia National Laboratory Tom Duffy, Princeton Chi-Chang Kao (not present), SLAC National Accelerator Center Martin Kunz, Lawrence Berkeley National Laboratory Mark Rivers, University of Chicago De-Ming Shu, Argonne National Laboratory Choong-shik Yoo, Washington State University

The Advisory Committee (AC) met on Monday, June 1 with HPCAT executive director (V. Struzhkin), management, (G. Shen, S. Sinogeikin), the HPCAT Executive Committee members, as well as HPCAT staff. The morning was devoted to presentations on current status as well as on a three-phase upgrade plan taking into account the possible upgrade of the Advanced Photon Source (APS) storage ring to a diffraction limited multi-bend achromat (MBA) "ultimate" storage ring. The Advisory Committee then discussed the following charges in the presence of Guoyin Shen and members of the HPCAT Executive Committee.

1. HPCAT facility – capabilities and competitiveness in the world;
2. HPCAT operation – productivity and quality;
3. HPCAT overall direction – future outlook;
4. HPCAT near future development projects (next 5 years) – advancement and reasonableness;
5. HPCAT upgrade plan with MBA lattice – preparedness and potential impacts.

A brief summary of the AC's assessment regarding these topics was presented at the end of the meeting and is repeated at the beginning of each topical section.

The Advisory Committee was generally impressed by the performance of HPCAT which operates four beam lines simultaneously at highest level. This is remarkable given the complexity and variety of different experimental techniques offered, and the excellent overall quality reflects positively in the publication record. The work-planning process for user experiments is very well organized, e.g., a local contact assigned to every user experiment and additional support is available during the actual beam time. This results in an efficient distribution of the workload among the scientific staff and gives individual staff members the opportunity to collaborate with various user groups. The committee is especially impressed by the staff performance in view of lower staffing levels than found at beam lines with comparable focus and performance, e.g., at international high-energy storage rings such as the ESRF and SPRing-8.

The three-phase upgrade developed by HPCAT provides a very sensible path to technical improvements that are required to address long-standing as well as novel problems in high-pressure science, e.g., precise equations of state and melting temperatures, electronic properties of H/Li/Na, strain-rate dependences, poly-amorphism is glasses and liquids, static pressures in excess of 5 Mbar.

1 Capabilities of facility and competitiveness in the world

Summary: The technical capabilities are world class and truly impressive. Several developments are world leading and can be expected to be followed by others. Achieving a facility of this quality would not have been possible without 15 years of consistent funding.

HPCAT operates four beam lines simultaneously at highest level. A second undulator in a canted arrangement was recently added to support two completely independent ultra-high brilliance beam lines. One branch is dedicated to flux-hungry spectroscopies such as x-ray emission (XES), inelastic (IXS), and nuclear resonant (NRS). The other branch is highly optimized for micro-diffraction studies with environmental support such as Laser heating and cryogenic cooling. The bending magnet emission fan is spatially split and delivers x-rays for two stations. One of them is dedicated to x-ray absorption spectroscopies and micro-diffraction, the other station offers white-beam Laue and energy-dispersive x-ray diffraction. All beam lines feature micro-focusing to accommodate studies on very small samples.

With these excellent experimental capabilities crystal structures and electronic states of matter are characterized under extreme conditions of pressure and temperature. The quality of the resulting data are superb and largely justify a world-class status of HPCAT as an experimental high-pressure facility. The study of phase-transition kinetics under rapid compression and de-compression as well as under conditions of modulated Laser heating is a novel and potentially ground-breaking addition to HPCAT's capabilities. This particular aspect can certainly be considered world-leading. At present the development of time-resolved studies reaches time-scales down to milli-seconds, but it appears that micro-second time scales are attainable with appropriate upgrades.

The development and successful operation of such a multitude of experimental capabilities was possible through stable and dedicated financial support over the last 15 years. The Advisory Committee hopes that changes in the funding structure for HPCAT with DOE-NNSA now providing 75 % of funds for HPCAT operations would have no discernible negative impact on the HPCAT facilities capabilities and staffing.

2 Operation – productivity and quality

Summary: HPCAT has highly qualified and motivated staff, an excellent publication record, and a high level of technical development. The high number of innovative projects could influence the balance between R&D and user support. This balance should be carefully monitored to maintain a high level of user satisfaction.

HPCAT has built and maintains are highly effective support structure for user experiments. Highly qualified and motivated staff and an extensive selection of support equipment and laboratories are key ingredients for the high quality of HPCAT's scientific productivity. The user experience at HPCAT is notably enhanced by a local contact person, high reliability of beam line optics and support equipment, and robust instrumentation in accessible areas. The local contact is responsible for smooth interaction with scheduled users including communication and discussion of experimental procedures prior and after an experiment. During the experiment the entire HPCAT staff is available to support the successful execution of the experimental plan. This results in an efficient distribution of the workload among the scientific staff and gives individual staff members the opportunity to collaborate with various user groups. Backup equipment for critical beam line components and computing resources significantly shortens potential downtimes caused by equipment failure.

The personal safety of users and staff as well as operational safety are well managed through interlock systems, engineered feedback systems, and clearly spelled out specifications for particular experimental setups.

Beam time allocation is distributed between HPCAT partners (60 %), the general APS user population (25 %), and internally for beam line readiness and R&D (15 %). The number of users has been relatively constant over the last six years indicating a certain level of maturity. Nevertheless the number of refereed publications has steadily increased over the same time period to about double the number in 2009 indicating a steady increase of productivity. The publication record is supplememented with other outreach activities such as the organization of scientific meetings, summer schools, and the APS-local high-pressure interest group.

In light of these developments, the committee is especially impressed by the staff performance given the somewhat lower staffing levels than found at beam lines with comparable focus and performance, e.g., at international high-energy storage rings such as the ESRF and SPRing-8. HPCAT recently engaged in several innovative projects with potentially great impact on high-pressure science. In light of this additional activity, an impact on productivity and user satisfaction (which is very high) may be unavoidable at similar staffing levels.

3 Overall direction – future outlook

Summary: The synergistic approach of HPCAT has been successful to provide excellent scientific results. Additional techniques such as single-crystal or multi-grain diffraction probably require additional effort in software development and user training to attract a broad user base and potentially replace powder diffraction.

HPCAT provides a large variety of x-ray scattering techniques to its users. This multi-pronged approach to address scientific problems has been very successful over the years. A recent special collection of papers published in RSI describes the status-quo at HPCAT as well as new developments. In this context, HPCAT has added new capabilities such as single-crystal or multi-grain diffraction to its expanding portfolio of experimental techniques. These developments are very exciting because they could replace the very popular powder diffraction technique for exceedingly small x-ray focal spot sizes, e.g., incase of the MBA upgrade.

For HPCAT, it seems natural to strive for an even larger portfolio of experimental techniques to continue this line of success. As HPCAT also realizes this expansion process, even though desirable for many users, is resource limited. For example, the replacement of powder diffraction with equivalent or superior techniques requires not only instrumental development, but also the creation and testing of evaluation software and very importantly the training of users in efficient use of the new techniques.

4 Near future development projects (next 5 years) – advancement and reasonableness

Summary: We agree with the three-phased upgrade of HPCAT, and all presented plans are very reasonable.

The emphasis on x-ray detectors in Phase-I of HPCAT's upgrade plan is a great way to optimize data collection but also to explore new applications such as time-resolved micro-diffraction. Improved focusing optics can provide spot sizes sufficiently small for scanning of samples in diamond-anvil-cells. In either case, new scientific opportunities will emerge, and HPCAT has developed a vision that creates connections between instrumental developments and scientific problems.

Technical progress (fast efficient detectors, more precisely machined mirrors and capillaries, new compression devices) leads to enhanced instruments that are then integrated into a new capability to benefit the user base and high-pressure science. HPCAT advances this dynamic process of interwoven areas very well with an eye on the needs and requests of their users and funding agencies.

5 Upgrade plan with MBA lattice – preparedness and potential impacts

Summary: Even though HPCAT is quite well prepared to address the improvements and changes expected from an APS upgrade to a low-emittance MBA lattice, we would need to hear more evidence about resulting transformational science, for example, from coherent diffraction or imaging approaches.

The present upgrade plan of the APS is driven by a change of the magnetic lattice of the storage ring to a multibend achromat (MBA) design. The implementation of the MBA is expected to reduce the horizontal electron emittance significantly. This should then lead to x-rays with much smaller divergence emitted by a smaller source, i.e., a vastly increased brightness leading to a significantly more coherent beam. With present undulators, the x-ray flux would be similar or even reduced because of the lower electron energy in the storage ring (6 GeV instead of todays 7 GeV). This situation can potentially be improved by installing superconducting undulators with shorter magnetic period than todays devices. The on-sample flux however could increase substantially if focusing devices captured a larger fraction of the x-ray beam than at present. In summary, the major change resulting from the MBA upgrade would be a smaller, more coherent x-ray beam.

HPCAT projects that the MBA upgrade is realized in about six years from today and has developed a specific upgrade plan with activities starting about three years before the MBA upgrade. This is a reasonable starting point in time which however may recede determined by the APS upgrade schedule. The MBA related upgrade appears to be Phase-II of HPCAT's overall development strategy. Phase-II emphasizes the improvement of beam delivery optics to adjust to the x-ray beam quality and, in particular new focusing elements to take advantage of the higher brilliance of the x-rays. These are very sensible steps in the MBA scenario.

Under the MBA upgrade plan of APS, bending magnets are replaced with 3-pole wigglers that deliver significantly more x-ray flux at energies above 20 keV. In HPCAT's Phase-II plan, this situation is exploited with a re-design of the bending magnet beam lines emphasizing high-energy micro-diffraction of solids and liquids which would be a very fruitful development.

In Phase-III of HPCAT's upgrade plan, the introduction of new x-ray techniques is envisioned. Such techniques presumably require a degree of coherence that is not available today but would be provided by an APS storage ring with the MBA-lattice. The addition of coherent diffraction and/or x-ray imaging to HPCAT's portfolio sounds intriguing. Here the Advisory Committee encourages HPCAT to further study the nature of these techniques in the framework of high-pressure science. Any evidence that transformational science could result would clearly strengthen the case of this aspect of HPCAT's upgrade plan.

APPENDIX 6.3: 2013 (Latest) Review Report by the Advanced Photon Source

Report of HP CAT Review March 5, 2013

Submitted June 21, 2013

Panel:
Andrew Campbell (University of Chicago, Department of Geophysical Sciences)
Serge Desgreniers (University of Ottawa, Physics Department)
William Stirling (APS SAC Chairman, CEA Grenoble DRT)
Glenn Waychunas (APS SAC member, Lawrence Berkeley National Laboratory, Earth Sciences Division

Brief Synopsis

HPCAT operates two beamlines (16-ID and 16-BM) at the APS dedicated to high pressure research encomposing materials, solid state and geophysical sciences. The main membership consists of the Carnegie Institution of Washington, Lawrence Livermore National Laboratory, Los Alamos National Laboratory, Sandia National Laboratory, University of Nevada at Las Vegas, and CDAC (a consortium of universities with interests in high pressure research). Funding has been stable at approx. 3M$/yr of which 75% comes from DOE-NNSA via Carnegie, and 25% from DOE-BES. General user proposal (GUP) time is 25%.

Scientific operation is very impressive with roughly 80 publications per year including many in high impact journals, a similarly impressive distribution of users from around the world, and several very important scientific findings over the review period. The facility has a remarkably broad range of techniques represented, many available in synchrony at a single beamline. The facility is competitive with analogous sets of beamlines at other world facilities, but has a unique integration of capabilities. Additionally, HP-CAT has an outstanding technique development record, which is closely connected with a noteworthy ability to train new scientists in high pressure synchrotron science.

Staffing appears adequate to continue to achieve at these levels, but there is evidence of strain, especially in consideration of new initiatives, and there is an issue with individual career development, for which no independent beam time for research is currently allotted. Management is through an executive committee that makes all critical decisions. A scientific advisory committee, which meets and is appointed at the discretion of the executive committee, seems to have a minor role, having met only twice over the last four years. Future plans, including the development of submicron beam capabilities, are very well coupled with the goals of the APS upgrade. Although challenging, they appear to effectively utilize existing expertise at other APS beamlines to good advantage. Other challenges include a better coupling to theoretical analysis involving several types of studies, and software development, which appears to trail new technique implementation in priority.

Detailed Analysis

Scientific Performance and Productivity

HP-CAT has produced impressive scientific research achievements over the review period, evidenced by a strong publication record, an outstanding production record for student theses, and many notable scientific findings. Some of the most significant science described during the review and/or contained in review materials supplied by the APS include: New descriptions of the ordered state in amorphous metals and carbon clusters (Science, 2012; Science, 2011); f-electron delocalization and volume collapse in metals (PRL, 2012; PRB, 2012); the reappearance at high pressures of superconductivity in iron arsenides (Nature, 2012); new complexity in the phase diagram and structural transitions in alkali metals (Nature Physics, 2011).

The publication record is among the best for any sector at the APS, averaging about 76 publications (journal articles, book chapters, student theses) a year, with the journal average alone (about 60 per year) comparing favorably with other strong sectors such as GSECARS. The number of theses (MS and PhD) average about 10 a year, which is exceptional. The fraction of publications in high impact journals is also noteworthy, with roughly 17% of journal articles in generally accepted high impact publications (based on APS-supplied information). Involvement in international conferences and workshops is significant, and appears to be in accord with the other high level scientific metrics.

Management Structure and Performance

Management of HPCAT is via an executive committee (EC) comprised of representatives of the main constituency. At present committee members include H.K. Mao (Executive director), R.J. Hemley (CIW), S. A. Gramsch (CDAC), Y. Zhao (UNLV), W. J. Evans (LLNL), N. Velisavljevic (LANL), W. Yang (HPSync) and the HPCAT director G. Shen. G. Shen replaced H.K.Mao as HPCAT director in 2009, while H.K. Mao directs the EC. The EC meets at least 4 times annually with the HPCAT director to arrive at operational and strategic decisions.

A HPCAT advisory committee, appointed by and holding meetings at the discretion of the EC, advises on scientific plans, development feasibility, and enhancements of existing capabilities. This committee has met only twice in the past four years, and does not appear to function with a purview or charge akin to the SAC for the APS, or as advisory groups do for other CATS. Current advisory members are C.C. Kao (SLAC), M. Rivers (CARS), W. Sturhahn (Caltech), M. Kunz (LBNL) and D. Shu (ANL). The role of this committee was discussed with several of the current members.

Funding for HPCAT has been consistent over the review period with a small increase. 75% of funds are derived from DOE-NNSA and 25% from DOE-BES, although post October 2012, the 75% from NNSA comes directly via an agreement with CIW, whereas prior to this date the NNSA funding came via the three NNSA-supported partners (CDAC, UNLV, LLNL). The DOE-BES funding is via a separate grant to CIW.

Performance of management is judged by reference to several criteria treated separately: use of APS-supplied beamtime; general user satisfaction; capabilities and sector development; future planning; adequacy of funding; staffing

APS beamtime usage

The four stations at HPCAT are fully utilized, with up to 43% oversubscription on 16-ID-B according to APS User Office supplied information. This is actually a low oversubscription rate for the APS as a whole. However information supplied by HPCAT conflicts with the User Office information and suggests more expectable to very high oversubscription rates, probably as a result of differences in the calculation using the same base numbers, or from a different metric (e.g. use of requested time on a different basis). For example, 16-ID-B has approximately a 700% oversubscription rate from 2010-2013, while the other stations have from 150% to 360% oversubscription rates. It is recommended that the actual general user subscription rates be recalculated by the User Office and normalized to the same metric used with other CATs. Discussions with staff indicate that the beamline stations are heavily used, and tight schedules must be maintained to afford reasonable fractions of time for development and commissioning of facilities.

General user satisfaction

The technical and scientific staff appears highly knowledgeable and very responsive to diverse users' requirements, and particularly to general APS users. Users seem quite satisfied with the data obtained from the HPCAT end stations, both in terms of quantity and quality. The APS USER Office supplied feedback from approx. 144 users who had submitted end-of-experiment (EOE) reports on their experiences, about 70 of whom made specific comments, all very complementary to the staff, and to the quality of the facilities. The proportion of users indicating successful or highly successful runs at HPCAT was about 80%, with the remaining fraction almost universally indicating partially successful runs. The apparent time between data acquisition and publication was very reasonable, also consistent with an organized and well-supported user program.

Current Capabilities and sector development

HPCAT has pioneered the concept of "integrated synchrotron radiation experiments" dedicated to compression science and technology to study matter at extreme conditions. X-ray probes were conceived, developed, and implemented to enable the studies of physical properties of dense matter. Structural, vibrational, electronic, and magnetic properties can be probed at high pressure and high/low temperature using the instrumentation housed in the HPCAT end-stations.

The experimental capabilities are spread over four different beam stations at Sector 16. Each station is equipped with different end-station capabilities. Two stations take advantage of a canted undulator source providing very high brilliance in the 5-40 keV range. The high brilliance ID beamline serves four different end-stations for x-ray spectroscopy, i.e., x-ray emission spectroscopy (XES), inelastic x-ray scattering (IXS), nuclear resonant inelastic x-ray spectroscopy (NRIXS) at stations ID-C/D/E, and micro-

diffraction at high pressure and high/low temperature at station ID-B. Two other stations make use of a split x-ray beam arising from the bending magnet source (BM). At the BM three end-stations allow for diverse scattering and spectroscopy experiments: micro-diffraction (both powder and single crystals) and x-ray absorption near edge structure (XANES) spectroscopy at stations BM-C/D and white beam Laue diffraction and large volume x-ray scattering at station BM-B. Whereas the BM stations accommodate experiments carried out at modest and high pressures, using diamond anvil and large volume Paris-Edinburgh pressure cells, and variable temperature using cryocooling or resistive heating of samples at high pressure, the ID stations are mostly used for experiments at extreme pressure and temperature conditions, with samples in diamond anvil cells probed by x-ray beams at small scale (less than 10 mm) and at pressures exceeding 100 GPa with simultaneous temperatures ranging from 5 to 5000K using cryostats or laser heating. HPCAT has played a leading role in developing techniques to probe samples at extreme conditions with synchrotron radiation beams.

It is obvious that considerable effort is devoted by staff to optimize synchrotron beam usage, and enhance the general user's experience. Hardware, data acquisition software and graphical user interfaces, and data analysis software allow rapid and efficient gathering of high-quality data. As added user's support, HPCAT also provides ancillary equipment located both at the beamlines and in adjacent laboratories, e.g., spectroscopy systems for pressure measurement and Raman scattering, cryostats, and pressure cell preparation and control, and sample loading equipment. The suite of experimental capabilities available at the HPCAT also is enhanced by other complementary capabilities offered at several other APS beamlines which interact with the HPCAT staff (e.g. GSECARS).

It is thus our finding that the HPCAT Facility at the Advanced Photon Source represents a world-class platform for condensed matter science at extreme conditions. The Facility offers a remarkable suite of scattering and spectroscopy experiments utilizing hard X-rays from both bending magnet and insertion device sources. At present, nowhere else in the world does such a plethora of x-ray scattering and spectroscopy instrumentation devoted to high pressure science exist "under one roof". In this respect, and for the quality of its installations, HPCAT is to be considered the leading synchrotron radiation facility dedicated to science at extreme conditions.

Future Directions

The principal research directions presented by the HP-CAT Director, Prof G. Shen, are:

- Reaching 0.5 TPa and beyond – structure, bonding and thermodynamic properties
- Time-dependent transformations and off-Hugoniot processes
- New materials – discovery and applications
- Novel states of matter and new chemistry
- Deformation and transport properties of materials
- Liquids and amorphous materials
- New technologies and instrumentation

These topics summarize the current HP-CAT program while pointing the way to new capabilities and novel science, exploiting the new opportunities offered by the APS Upgrade and by the ongoing program of beamline technique development. It is noteworthy that the investigation of materials under extreme conditions is a major topic of the APS Upgrade, and hence that the HP-CAT programs are well in phase with the plans for the APS as a whole.

Phased upgrade program

The beamline management has developed a phased Upgrade plan, with Phase 1 currently underway (2012-2014); Phase 2 will follow (2014 – 2016), with Phase 3 scheduled for 2015/2016 – 2017. Phase 1 involves installation of a canted undulator, as well as time-resolved optics and advanced detectors. Phase 2 is synchronized with the APS Upgrade, leading to sub-micron probes and nanometer resolution X-ray imaging. For Phase 3, more work on advanced detectors is planned with further development programs on the imaging capabilities of the CAT (fast X-ray microscopy and tomography, and high-pressure coherent diffraction imaging.)

We find this phased program to be sensible and coherent, building on the experience and expertise of the beamline team, while exploiting the improvements brought about by the APS Upgrade (and employing APS expertise where necessary). The sub-micron focusing (a world first for a high pressure beamline) and nanometer resolution imaging will permit "next generation" experiments, including

- Even more extreme p-T conditions (towards the 10^4 K / TPa regime)
- More precise determinations
- Single-crystal experiments at the sub-micron level
- Grain boundary studies
- Combinatorial studies
- Composite materials

Technique development and sample environment

As mentioned above, a number of instrument/technique developments are planned for the next few years, continuing with the impressive program of equipment and technique development at HP-CAT. The result of this work over the last few years is a facility with exceptional integration of many different experimental techniques. The very successful laser-heating development will be continued, focusing on fast temperature changes to allow time-resolved studies. Single-crystal X-ray diffraction at high pressures is a very promising method. For time-resolved investigations the "white" beam Laué technique should allow studies of dislocations and deformations under pressure and stress. Another white beam method under development is very high speed radiography (up to 10,000 frames per second), used to study the dynamics of liquids at high pressures. For inelastic scattering a significantly smaller focus (5x5 μm^2 or even 1x1 μm^2) is planned.

The new science that will become possible depends to a large extent on the remarkable sample environment equipment made available by the beamline team. Sample

environment is a particular strength of this CAT. New work is planned in the three major areas, namely:

- Expanding capabilities in p, T and t (crysotats, diamond anvil cells(DAC), heated DACs, laser heating, dynamic pressure control)
- Sample preparation and probes (on- and off-line ruby systems and Raman systems)
- Increased efficiency of beamtime use (membrane and mechanical remote pressure control, on-line pressure measuring)

Taken overall, the CAT management and beamline staff have presented a coherent program of development over the next few years which is in phase with the planned APS Upgrade and should provide new scientific opportunities, while maintaining the CAT's position as one of the world's very best (if not the best) high pressure beamlines.

Staffing

Current staffing consists of 7 beamline scientists (100% funded), 3 beamline associates (100% funded), 3 support staff (100% funded), 3 postdocs (one 100% funded, and 2 others funded at 50% by the HPCAT operational fund). This yields effectively, 15 full time staff supported by the CAT.

Overall staffing was evaluated through discussions with staff both in a group setting, and privately, and by comparisons with staffing at other beamlines and CATS at APS and elsewhere. Although the staffing appears appropriate on a station/staff ratio basis, given the throughput of the beamlines, the number and complexity of the techniques that are supported, the amount of development in progress, and the aggressive nature of the coming upgrades, it appears the staffing may fall short of what is desirable.

Several staff members indicated they have heavy workloads and little time for their own intellectual and scientific development (in part due to HPCAT not providing staff priority research time). Others indicate that the demands of technique and software development place much stress on them, without elasticity in the organization to provide for this (e.g. with student or tech support, or acquisition of additional support staff). Not all staff had these concerns, but these considerations appear to have more significance at HPCAT than other CATs recently reviewed.

Staffing number and workload issues reflect an understandable tension between the need to drive relentlessly forward in the achievement of a world-class facility, and the individual needs of staff for a satisfying work environment that affords career-development potential. A challenge for HPCAT and for the APS in general is how to mitigate this tension for the good of all, so that significant numbers of staff are not disadvantaged. This is a crucial issue for the acquisition of new beamline scientific staffing, and for the ultimate encouragement of young scientists to enter such careers.

Adequacy of Funding

Funding for HPCAT has been consistent over the review period with a small increase. 75% of funds are derived from DOE-NNSA and 25% from DOE-BES, although post October 2012,

the 75% from NNSA comes directly via an agreement with CIW, whereas prior to this date the NNSA funding came via the three NNSA-supported partners (CDAC, UNLV, LLNL). The DOE-BES funding is via a separate grant to CIW. The principle issue with this support is that it is not diversified, and hence is subject to large potential changes. This is likely more a concern with the large block of NNSA funding. Given that the NNSA work is not subject to this review, and cannot be completely revealed, it is not possible to judge where this programmatic funding may be headed in longer term. As this is an appropriate concern for the APS as a whole, it is recommended that the management keep the APS informed about any potential changes in NNSA support well in advance.

Advisory Structure

The HPCAT scientific advisory committee, appointed by and holding meetings at the discretion of the HPCAT executive committee, advises on scientific plans, development feasibility, and enhancements of existing capabilities, at least in concept. However this committee has met only twice in the past four years, and does not appear to function with a purview or charge akin to the SAC for the APS, or as advisory groups do for other CATS. Current advisory members are C.C. Kao (SLAC), M. Rivers (CARS), W. Sturhahn (Caltech), M. Kunz (LBNL) and D. Shu (ANL). The role of this committee was discussed with several of the current members, who suggested it is not being used to advantage by HPCAT, or in a proactive way to address some of the challenges facing the facility. It would seem that a more regular meeting schedule for this committee, including significant charges and review, would benefit HPCAT, both in terms of addressing existing challenges, and also helping to remove the appearance that HPCAT largely operates without external oversight.

Self-identified ongoing challenges

Management identified several areas of significant challenges: Two major ones are software development and theoretical interconnection. Because of the large number of supported technologies, as well as their continuous development, and the workload of the staff, software development of user interfaces and analysis software often falls behind technical capability. This is a challenge for all APS CATS, but especially so for HPCAT due to the large number of different supported methodologies. The crux of the challenge is for management to decide how to allocate staffing to support the technical operation while keeping software development at pace. For unique staff scientists who have gifted programming skills, moving between these areas is facile. However in most cases specialists in software development are needed, and are much more efficient than staff not dedicated to such efforts. This is an area where close interaction and advice from the advisory committee could be very beneficial, especially in planning for the upgrade and resulting needs.

On the theoretical side the most likely enhancements might come from improvement and nurturing of increased connections with theoreticians working on materials or classes of phenomena being studied at HPCAT. This is another area where the advisory committee could be of great help, especially if appropriate theorists are added. An improved interconnection with theorists would be beneficial in support of present research

directions, but it would also be useful in identification of potential rich pathways for future investigations and technique/capability enhancements.

Other Challenges

We have indicated the challenges with staffing earlier, but another potential issue is the "insider" nature of the CDAC program. CDAC members are encouraged to submit proposals to the general user program, but also have a direct access route through the CDAC program to beamtime. It is not completely clear that this arrangement benefits the CDAC program or the general user program. On the one hand few CDAC members receive time in the general user program due to oversubscription and intense competition, so it is not clear that this affords useful experience to CDAC members, while it adds competition for "outside" general users. On the other hand CDAC members seem to have most of their needs serviced within the CDAC programmatic beamtime. It is in HPCATs best interest to contribute to a very strong and demonstrably open general user program at APS, and hence make available as much general access as possible. Hence for all these reasons CDAC application to general user time seems unwarranted.

An additional consideration is that individual staff research time, when not available during technique commissioning, is only available through staff application for general user time. This sets up a potential competition between general users, CDAC users and HPCAT staff for the same beamtime allocations.

APPENDIX 6.4: List of Individual Users in 2015

First Name	Last Name	Position	Affiliation
Christopher	Adcock	Post Doc.	University of Nevada, Las Vegas
Muhetaer	Aihaiti	Research Scientist	Carnegie Institution of Washington
Andrew	Alvarado	Graduate	University of Nevada, Las Vegas
Bruce	Baer	Research Scientist	Lawrence Livermore National Laboratory
Ligang	Bai	Post Doc.	Carnegie Institution of Washington, HPCAT
Jason	Baker	Graduate	University of Nevada, Las Vegas, Department of Physics and HiPSEC
Maria	Baldini	Research Scientist	Carnegie Institution of Washington
Courtney	Bartlett	Graduate	University of Nevada, Las Vegas
Joel	Bernier	Research Scientist	Lawrence Livermore National Laboratory
Venkata	Bhadram	Post Doc.	Carnegie Institution of Washington
Wenli	Bi	Research Scientist	University of Illinois
Jasmine	Bishop	Undergraduate	University of Utah
Reinhard	Boehler	Research Scientist	Carnegie Institution of Washington
Anna	Boehmer	Post Doc.	Ames Laboratory
Dmitry	Bolmatov	Post Doc.	Brookhaven National Laboratory
Gustav	Borstad	Post Doc.	Army Research Laboratory
Jodie	Bradby	Faculty	Australian National University
Keenan	Brownsberger	Undergraduate	Whitworth University
Pamela	Burnley	Research Scientist	University of Nevada, Las Vegas
Andrew	Campbell	Faculty	University of Chicago
Huawei	Chen	Graduate	Arizona State University
Yu-chen	Chen	Research Scientist	Brookhaven National Laboratory
Xuliang	Chen	Faculty	High Magnetic Field Laboratory, Chinese Academy of Sciences
Bijuan	Chen	Graduate	Institute of Physics, Chinese Academy of Sciences
Qiang	Chen	Graduate	University of Tennessee
Bin	Chen	Faculty	University of Hawaii at Manoa
Bethany	Chidester	Graduate	University of Chicago
Yongseong	Choi	Research Scientist	Argonne National Laboratory
Shengqi	Chu	Research Scientist	The Institute of High Energy Physics, Chinese Academy of Science
Lin	Chuanlong	Post Doc.	Carnegie Institution of Washington, HPCAT
Benjamin	Cochain	Post Doc.	ISTeP, UPMC
Sean	Collins	Undergraduate	Oakland University
Anthony	Connolly	Graduate	University of Nevada, Las Vegas
Jacob	Cooper	Undergraduate	University of Tennessee
Neala	Creasy	Graduate	Yale University
William	Cureton	Undergraduate	University of Tennessee

Hyunchae	Cynn	Research Scientist	Lawrence Livermore National Laboratory
Dana	Dattelbaum	Research Scientist	Los Alamos National Laboratory
Kierstin	Daviau	Graduate	Yale University
Anne	Davis	Undergraduate	Carnegie Institution of Washington
Shanti	Deemyad	Faculty	University of Utah
Przemyslaw	Dera	Faculty	University of Hawaii at Manoa
S.	Desgreniers	Faculty	U. of Ottawa
Matthew	Diamond	Graduate	University of California at Berkeley
Dawei	Dong	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Haini	Dong	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Vadym	Drozd	Research Scientist	Florida International University
Mihindra	Dunuwille	Post Doc.	University of Utah
Lars	Ehm	Faculty	Stony Brook University
William	Evans	Research Scientist	Lawrence Livermore National Laboratory
Chris	Fancher	Post Doc.	North Carolina State University
Greg	Finkelstein	Post Doc.	Caltech
Rebecca	Fischer	Graduate	University of Chicago
Zachary	Fussell	Undergraduate	University of Nevada, Las Vegas
Seth	Gainey	Graduate	University of Nevada, Las Vegas
Jennifer	Girard	Post Doc.	Yale University
Huiyang	Gou	Post Doc.	Carnegie Institution of Washington
Stephen	Gramsch	Research Scientist	Carnegie Institution of Washington
Tingting	Gu	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Kemin	Gu	Undergraduate	Center for High Pressure Science and Technology Advanced Research
Xiaofeng	Guo	Post Doc.	Los Alamos National Laboratory
Bianca	Haberl	Research Scientist	Oak Ridge National Laboratory
Katie	Ham	Graduate	University of Alabama at Birmingham
Fei	Han	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Elisabeth	Hausrath	Faculty	University of Nevada, Las Vegas
Jane	Herriman	Graduate	Caltech
Christopher	Higgins	Graduate	University of Nevada, Las Vegas
Binyang	Hou	Post Doc.	Carnegie Institution of Washington
Mingqiang	Hou	Post Doc.	Carnegie institution of washington
Dong	Hou	Graduate	North Carolina State University
Rostislav	Hrubiak	Post Doc.	Carnegie Institution of Washington, HPCAT
Qingyang	Hu	Post Doc.	Carnegie Institution of Washington
Yi	Hu	Graduate	University of Hawaii at Manoa
Dongyang	Huang	Graduate	Center for High Pressure Science and Technology Advanced Research
Daniel	Hummer	Post Doc.	UCLA
Daijo	Ikuta	Research Scientist	Carnegie Institution of Washington

Matthew	Jacobsen	Post Doc.	Los Alamos National Laboratory
Tomasz	Jaron	Post Doc.	Carnegie Institution of Washington
Wageesha	Jayasekara	Graduate	Iowa State University/Ames Laboratory
Jason	Jeffries	Research Scientist	Lawrence Livermore National Laboratory
Zsolt	Jenei	Research Scientist	Lawrence Livermore National Laboratory
Cheng	Ji	Post Doc.	Carnegie Institution of Washington, HPSynC
JIdong (Samuel)	Jiang	Research Scientist	Argonne National Laboratory
Xiujuan	Jiang	Post Doc.	Pacific Northwest National Laboratory
Shan	Jiang	Graduate	University of Western Ontario
Michael	Jugle	Graduate	University of Utah
Feng	Ke	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Jongjin	Kim	Post Doc.	Argonne National Laboratory
Minseob	Kim	Research Scientist	Washington State University
Donghoon	Kim	Graduate	Yonsei university
Jae-Hyun	Klepeis	Research Scientist	Lawrence Livermore National Laboratory
Byeongkwan	Ko	Graduate	Arizona State University
Yoshio	Kono	Research Scientist	Carnegie Institution of Washington, HPCAT
Karunakar	Kothapalli	Post Doc.	Ames Laboratory
Andreas	Kreyssig	Research Scientist	Ames Laboratory
Tyler	Kukla	Undergraduate	Northwestern University
Ravhi	Kumar	Faculty	University of Nevada, Las Vegas
Xiaojing	Lai	Graduate	University of Hawaii at Manoa
Barbara	Lavina	Research Scientist	University of Nevada, Las Vegas, Department of Physics and HiPSEC
Clemence	Leroy	Graduate	IMPMC - UPMC
Quanjun	Li	Research Scientist	Carnegie Institution of Washington, Jilin University
Rui	Li	Graduate	Jilin University
Liangliang	Li	Graduate	Center for High Pressure Science and Technology Advanced Research
Renfeng	Li	Graduate	Center for High Pressure Science and Technology Advanced Research
Wentao	Li	Graduate	Center for High Pressure Science and Technology Advanced Research
Nana	Li	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Kuo	Li	Research Scientist	Center for High Pressure Science and Technology Advanced Research
Bing	Li	Post Doc.	Carnegie Institution of Washington, HPSynC
Xiang	Li	Graduate	Penn State University
Zongyao	Li	Graduate	The University of Texas at Austin
Qian	Li	Undergraduate	University of Nevada, Las Vegas
Brian	Light	Graduate	University of Nevada, Las Vegas
Feng	Lin	Graduate	University of Utah
Jonathan	Lind	Post Doc.	Lawrence Livermore National Laboratory
Magnus	Lipp	Research Scientist	Lawrence Livermore National Laboratory

Haozhe	Liu	Faculty	Center for High Pressure Science and Technology Advanced Research
Fengliang	Liu	Graduate	Center for High Pressure Science and Technology Advanced Research
Fuyang	Liu	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Xiaobing	Liu	Post Doc.	Northwestern University
Jin	Liu	Post Doc.	Stanford University
Jin	Liu	Graduate	The University of Texas at Austin
Hongbo	Lou	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Xujie	Lu	Post Doc.	UNLV and LANL
Mary	MacLean	Undergraduate	University of Utah
Adam	Makhluf	Graduate	UCLA
Craig	Manning	Faculty	UCLA
Hokwang	Mao	Faculty	Carnegie Institution of Washington
Madeline	Margevicius	Undergraduate	Los Alamos National Laboratory
Daniel	Mast	Graduate	University of Nevada, Las Vegas
Andrew	Mattillion	Undergraduate	University of Chicago
Lisa	Mauger	Graduate	Caltech
David	McKenzie	Faculty	University of Sydney
Kenji	Mibe	Faculty	Earthquake Research Institute, Univ Tokyo
Vahe	Mkrtchyan	Graduate	University of Nevada, Las Vegas
Jamie	Molaison	Research Scientist	Oak Ridge National Lab
Jeffrey	Montgomery	Graduate	University of Alabama at Birmingham
Gregory	Myers	Undergraduate	University of Chicago
Jason	Nikkel	Graduate	North Carolina State University
Carole	Nisr	Post Doc.	Arizona State University
Ella	Olejnik	Post Doc.	University of Utah
Michael	Page	Graduate	Florida International University
Raul	Palomares	Graduate	University of Tennessee
Changyong	Park	Research Scientist	Carnegie Institution of Washington
Sulgiye	Park	Graduate	Stanford University
Jeffrey	Pigott	Graduate	Ohio State University
Michael	Pravica	Faculty	University of Nevada, Las Vegas
Fei	Qin	Graduate	Northwestern University
Benjamin	Rainwater	Graduate	Georgia Institute of Technology
Selva Vennila	Raju	Faculty	Florida International University
Mary	Reagan	Graduate	Stanford University
Nolan	Regis	Graduate	University of Nevada, Las Vegas
Xiangting	Ren	Graduate	Center for High Pressure Science and Technology Advanced Research
Brandon	Rhymer	Graduate	Stony Brook University
Dylan	Rittman	Graduate	Stanford University
Richard	rowland	Graduate	University of Nevada, Las Vegas, Department of Physics and HiPSEC

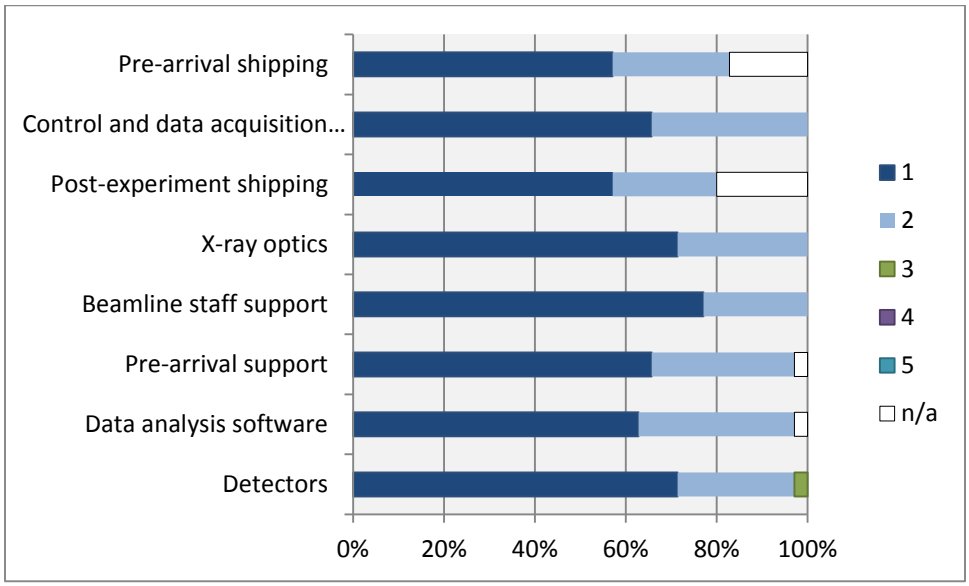
Young Jay	Ryu	Graduate	Washington State University
Gopi	Samudrala	Research Scientist	University of Alabama at Birmingham
Aashish	Sapkota	Graduate	Iowa State University/Ames Laboratory
Anne Marie	Schaeffer	Graduate	University of Utah
Henry	Scott	Faculty	Indiana University South Bend
Kelly	Seaborg	Undergraduate	University of Nevada, Las Vegas, Department of Physics and HiPSEC
Donghoon	Seoung	Post Doc.	SLAC National Accelerator Laboratory
Anat	Shahar	Faculty	Carnegie Institution of Washington
Hannah	Shelton	Graduate	University of Hawaii at Manoa
Yingxia	Shi	Graduate	Stanford University
Yuki	Shibazaki	Faculty	Tohoku University
Thomas	Shiell	Graduate	Australian National University
Sang-Heon	Shim	Faculty	Arizona State University
Nilesh	Shinde	Graduate	University of Western Ontario
Melissa	Sims	Graduate	Stony Brook University
Emily	Siska	Graduate	University of Nevada, Las Vegas
Thomas	Smart	Graduate	University of California at Berkeley
Jesse	Smith	Research Scientist	Carnegie Institution of Washington, HPCAT
Spencer	Smith	Graduate	University of Alabama at Birmingham
Quinlan	Smith	Graduate	University of Nevada, Las Vegas
Daniel	Sneed	Graduate	University of Nevada, Las Vegas
Maddury	Somayazulu	Research Scientist	Carnegie Institution of Washington
Yang	Song	Faculty	University of Western Ontario
Vincenzo	Stagno	Research Scientist	Ehime University
Timothy	Strobel	Faculty	Carnegie Institution of Washington
Viktor	Struzhkin	Research Scientist	Carnegie Institution of Washington
Fei	Sun	Graduate	IOP and HPSTAR
Wojciech	Tabis	Post Doc.	University of Minnesota
Lingyun	Tang	Post Doc.	Florida International University
Raman	Thiyagarajan	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Sally June	Tracy	Graduate	Caltech
Cameron	Tracy	Post Doc.	Stanford University
Phuc	Tran	Undergraduate	hipsec, unlv
Oliver	Tschauner	Faculty	University of Nevada, Las Vegas
Katlyn	Turner	Graduate	Stanford University
Rajasekarakumar	Vadapoo	Post Doc.	Carnegie Institution of Washington
Nenad	Velisavljevic	Research Scientist	Los Alamos National Laboratory
Yogesh	Vohra	Faculty	University of Alabama at Birmingham
Jeffrey	Walters	Graduate	University of Tennessee
Hsien-Hau	Wang	Research Scientist	Argonne National Laboratory
Qianqian	Wang	Post Doc.	Carnegie Institution of Washington
Junyue	Wang	Post Doc.	Carnegie Institution of Washington

Lijuan	Wang	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Yajie	Wang	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Lin	Wang	Research Scientist	Carnegie Institution of Washington, HPSynC
Jianwei	Wang	Faculty	Louisiana State University
Fei	Wang	Graduate	Northwestern University
Yuejian	Wang	Faculty	Oakland University
Shanmin	Wang	Post Doc.	University of Nevada, Las Vegas
Yonggang	Wang	Post Doc.	University of Nevada, Las Vegas
Liping	Wang	Research Scientist	University of Nevada, Las Vegas
Matthew	Ward	Post Doc.	Carnegie Institution of Washington
Erik	Watkins	Research Scientist	Los Alamos National Laboratory
Samuel	Weir	Research Scientist	Lawrence Livermore National Laboratory
Melanie	White	Undergraduate	UNLV High Pressure Science and Engineering Center
Yuming	Xiao	Research Scientist	Carnegie Institution of Washington, HPCAT
Fengping	Xiao	Graduate	University of Western Ontario
Shengyi	Xie	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Xiaolin	Xu	Graduate	Caltech
Cong	Xu	Graduate	Center for High Pressure Science and Technology Advanced Research
Xiaozhi	Yan	Graduate	Center for High Pressure Science and Technology Advanced Research
Liuxiang	Yang	Post Doc.	Carnegie Institution of Washington
Wenge	Yang	Faculty	Carnegie Institution of Washington, HPSynC
Hong	Yang	Graduate	Center for High Pressure Science and Technology Advanced Research
Hong	Yang	Undergraduate	Center for High Pressure Science and Technology Advanced Research
wenge	yang	Research Scientist	Carnegie Institution of Washington, HPSynC
FAN	YANG	Graduate	Stanford University
Jing	Yang	Graduate	The University of Texas at Austin
Howard	Yanxon	Undergraduate	HiPSEC, UNLV
Jianjun	Ying	Post Doc.	Carnegie Institution of Washington
A	Yoshinaka	Graduate	U. of Ottawa
Zhenhai	Yu	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Hongsheng	Yuan	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Zhidan	Zeng	Post Doc.	Carnegie Institution of Washington, HPSynC
Qiaoshi	Zeng	Research Scientist	Carnegie Institution of Washington, HPSynC
Eloisa	Zepeda-Alarcon	Graduate	University of California at Berkeley
Haidong	Zhang	Post Doc.	Carnegie Institution of Washington
Li	Zhang	Faculty	Center for High Pressure Science and Technology Advanced Research
Linji	Zhang	Graduate	Center for High Pressure Science and Technology Advanced Research

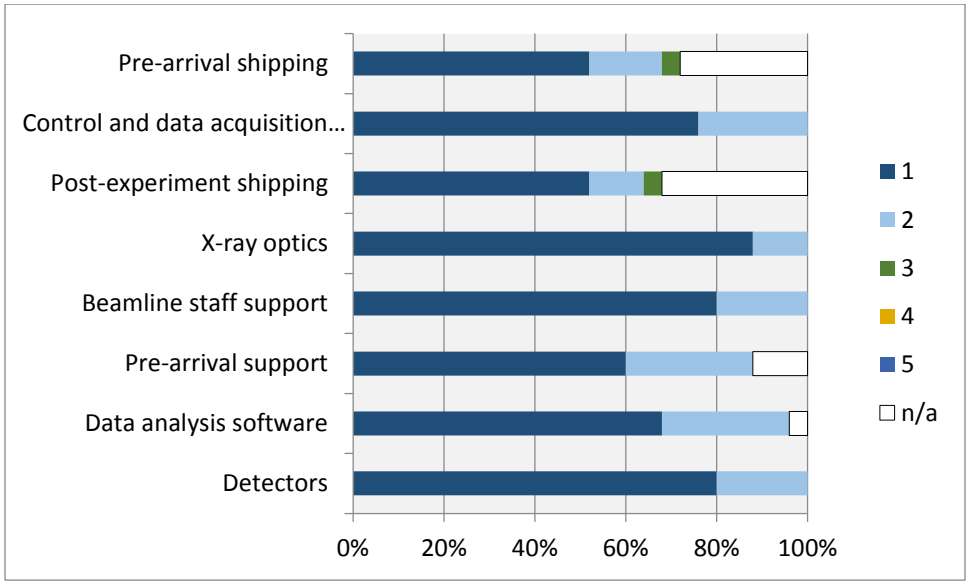
Ganghua	Zhang	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Ganghua	Zhang	Post Doc.	Center for High Pressure Science and Technology Advanced Research
Shuai	Zhang	Graduate	University of California at Berkeley
Jin	Zhang	Research Scientist	University of Hawaii at Manoa
Dongzhou	Zhang	Research Scientist	University of Hawaii/GSECARS
Fuxiang	Zhang	Research Scientist	University of Michigan
Zhisheng	Zhao	Post Doc.	Carnegie Institution of Washington
Yongsheng	Zhao	Graduate	Center for High Pressure Science and Technology Advanced Research
Chonghang	Zhao	Graduate	Stony Brook University
Mikhail	Zhernenkov	Research Scientist	Brookhaven National Laboratory
Jinlong	Zhu	Post Doc.	University of Nevada, Las Vegas
Kirill	Zhuravlev	Faculty	Moraine Valley Community College

APPENDIX 6.5: Users' Feedback after Experiments in 2014 and 2015

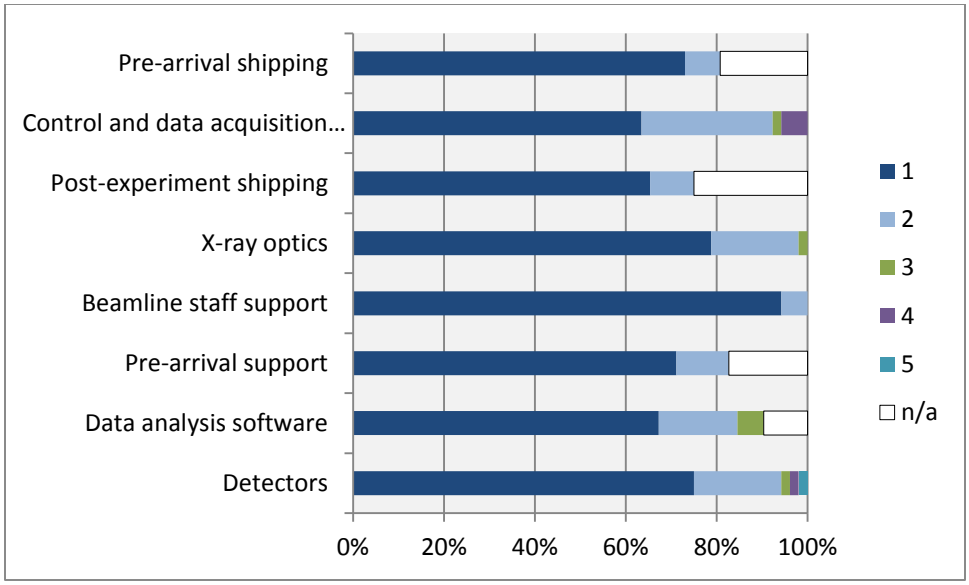
16-BMB – FY 2015



16-BMB – FY 2014



16-BMD – FY 2015



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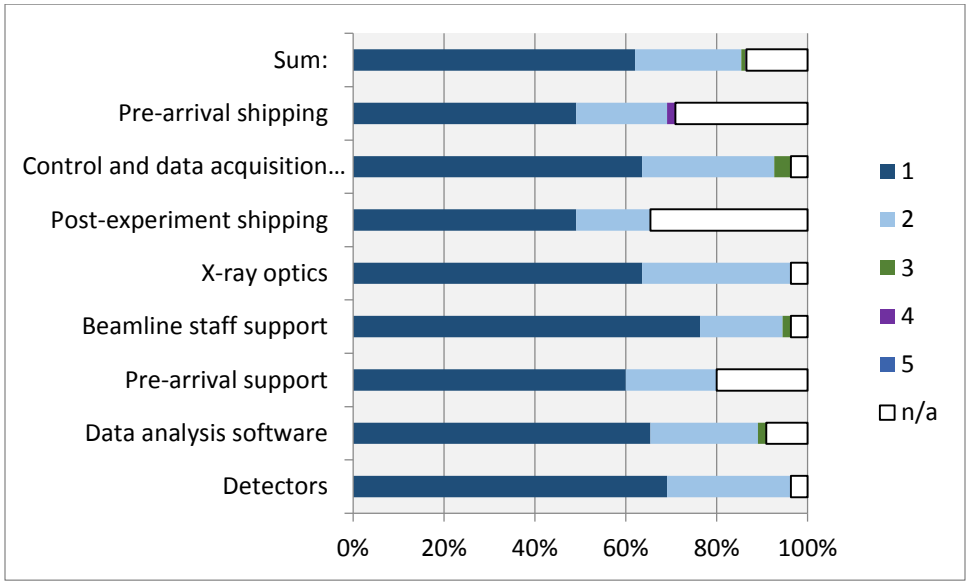
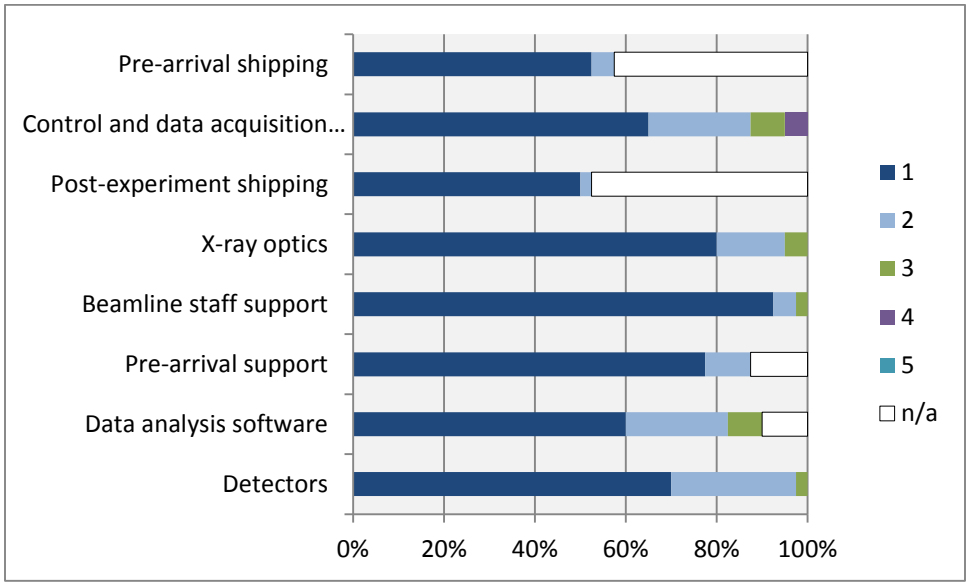
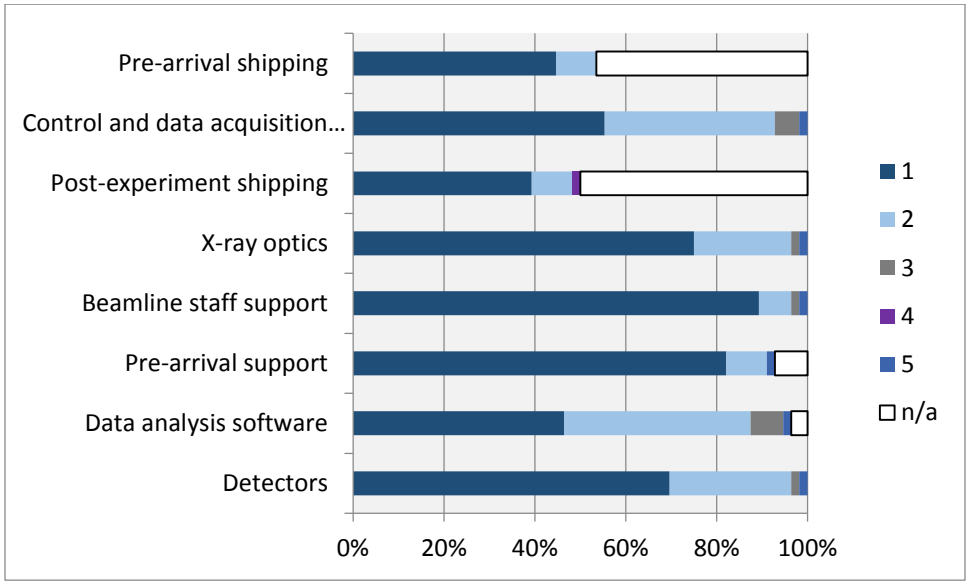


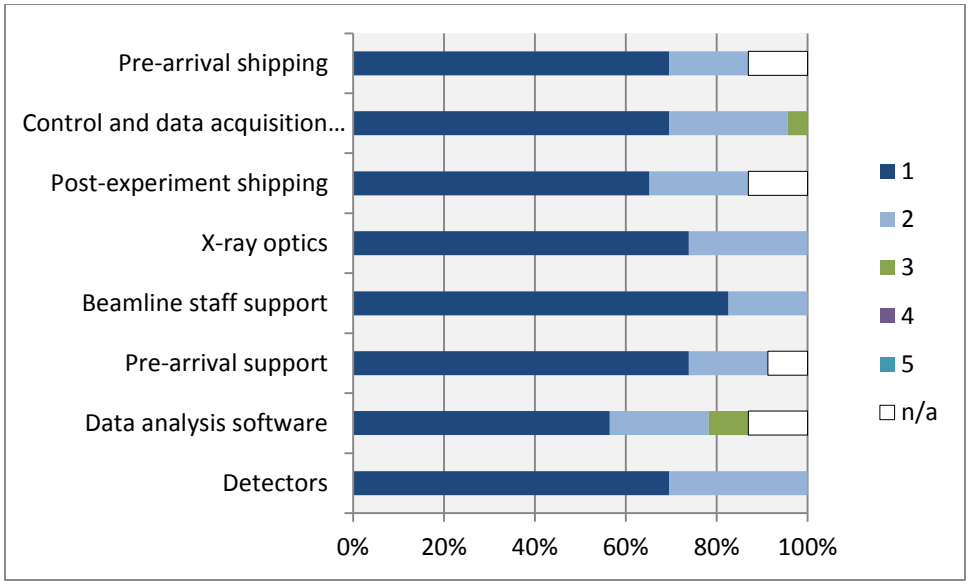
16-IDB – FY 2015



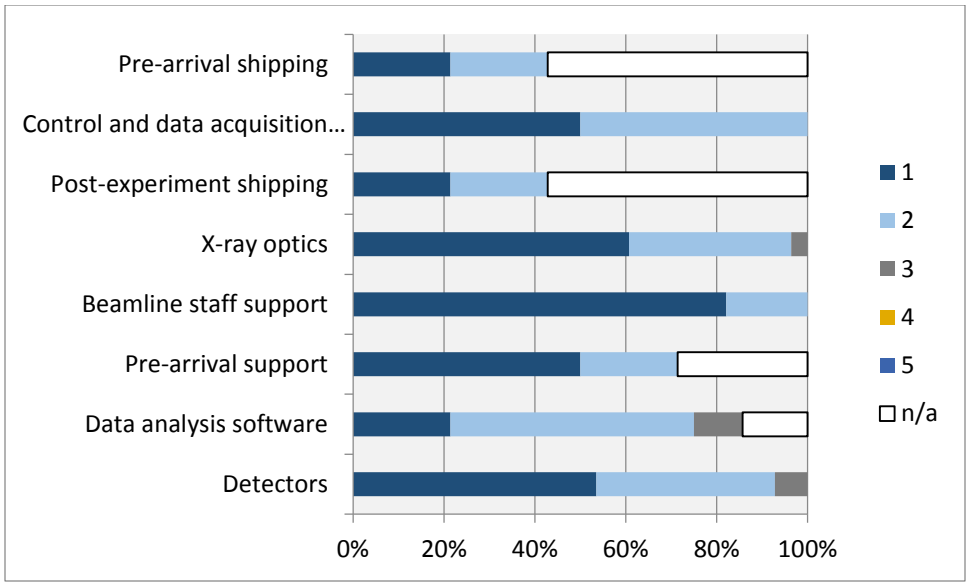
16-IDB – FY 2014



16-IDD – FY 2015



16-IDD – FY 2014



Some examples of users' comments in 2015 are listed below:

"*During this beamtime there was an improvement in the aligning software which was really important in increasing the efficiency of data acquision*." –Shanti Deemyad, Univ of Utah (IDB)

"*Technical support by the beam-line staff was excellent. We were able to achieve all our experimental objectives for this run*." – Yogesh Vohra, Univ of Alabama at Birmingham (IDB)

"*Overall very satisfied. For the "satisfied" parts - I would like to see video capture capability of sample heating. Currently you can monitor the camera during heating, but would be nice to be able to capture continue video during heating. Again, overall very satisfied. Thanks* " – Nenad Velisavljevic, LANL (IDB)

"*Great support from BL scientist and a tech. Special thanks to Curtis for his tremendous help*." - Mikhail Zhernenkov, Brookhaven National Laboratory (BMD)

"*I observed the program, blue diamond malfunctioning. It gives wrong scan plots few times. The communication through the network is still troublesome, I heard from the beamline staff.*" – Jae-Hyun Klepeis, LLNL (BMD)

"*X-ray acquisition software was buggy over the weekend*." – Dylan Rittman, Stanford (BMD)

"*The detector needs restarting once a while. No quite stable*." – Wenge Yang, CIW (BMD)

"*As always, the best support and help is provided from the beamline scientist and other staffs."* – Sulgiye Park, Stanford (BMD)

"*It is a new break through that low temperature and high pressure study of amorphous sulfur in Paris Edinburg Cell.I have some suggestions.Firstly,at low temperature and higher pressure study of amorphous sulfur is need to be improved, althrough it is difficult(Last time, highest pressure is 4GPa,at 80K). Secondly, at higher temperature and high pressure study of liquid sulfur is difficult because liquid sulfur is easy to flow out.Maybe, it is necessary that liquid materials study of high temperature and high pressure needs new assembling.*" – Linji Zhang, CIW (BMB)

"*Beamline scientist Dr. Yoshio Kono provided excellent support and all our planned experiments were successful*." – Yogesh Vohra, UAB (BMB)

"*I am really satified with this experiment. The beamline staff here are very professional, kind and patient. Hope to do more experiments here in the near future.*" – Hongsheng Yuan, HPSTAR (BMB)

"*This run, as compared to previous work, we required a lot more beamline support. I would like to say that as always beamline staff (including computer support Arun, and offline staff Kurtis and Eric) were exceptionally helpful - these guys are truly dedicated! As far as experiments, improvements have been made with addition of pilatus vs point detector for signal analysis and that is a great advantage. Overall a high satisfaction rating."* – Nenad Velisavljevic, LANL (IDD)

"*Yuming is consistently so helpful.*" – Mary Reagan (IDD)