ANL-24/41

Report on the HP-CAT Workshop-2023 held at the Advanced Photon Source

X-ray Science Division

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prepared by:
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X-ray Science Division, Argonne National Laboratory

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The conceptual framework and the founding partner and institutional agreements for the High Pressure Collaborative Access Team (HP-CAT) were established in late 1990's. In the following years, core infrastructure of HP-CAT was built at sector 16 of the Advanced Photon Source (APS) and the first x-ray light was delivered on July 19, 2002 at 7:00pm – followed by an official commemoration ceremony on July 26, 2002.

Over the ensuing years, HP-CAT grew into a premier facility dedicated for synchrotron-based x-ray measurements of materials under extreme pressure conditions. For 20+ years now, HP-CAT has been recognized as a world leader in synchrotron x-ray sciences and developments that have made significant impact in our understanding of materials at extreme conditions. From a single beamline in 2002, HP-CAT grew in scope and infrastructure and ultimately becoming the largest dedicated synchrotron high-pressure research facility in the world. HP-CAT facility consists of nine stations (five experimental and four optics stations), as well as an extensive off-line laboratory space. Over this period, there was also a significant growth in user base and the array of capabilities substantially evolving to meet the research needs of the user community and pushing the frontiers of high-pressure research. The array of capabilities across the multiple simultaneously operational experimental stations allows for generation of extreme pressures (from ambient to >500 GPa), temperatures from ~ 4K to 4000+ K, strain rates up to $\sim 10^3$/s, and various complex loadings, and all of the above coupled with a multitude of in situ probes that include x-ray diffraction, imaging, inelastic scattering and spectroscopy.

At the heart of HP-CAT is the user community. From a core founding-partner group in 1999, HP-CAT grew to over 850 user visits per year from worldwide high-pressure research groups. The 100+ annual publications and over 130 graduate Ph.D. theses supported to date resulting from work done at HP-CAT is a true testament to the strength of our community, partnerships, and visionary direction dating back to initiation of HP-CAT in 1999. HP-CAT's Mission is to advance scientific understanding of matter under extreme conditions through the development of state-of-the-art synchrotron x-ray capabilities and high-pressure apparatus – and our mission will remain as we continue to advance.

With the organization of this workshop, and this subsequent workshop report, our goal is to bring together the HP-CAT community to celebrate the 20-year anniversary of operations, to share in the numerous achievements – which we as a community have achieved over the years – and to look ahead at the exciting opportunities and challenges on the horizon. Over the last five years, APS has been preparing for a significant upgrade to the storage ring and associated components. The $850M project, which leverages over $1.1B existing infrastructure, commenced on April 17, 2023, and is expected to complete in one year. The comprehensive APS upgrade (APS-U) includes replacement of the original storage ring with a new multi-bend achromat (MBA) lattice that will result in a brightness increase by orders of magnitude and an even more significant increase in coherence. With particular reference to HP-CAT, and the high-pressure research field in general, the gain from upgrade is a smaller beam with orders of magnitude increase in on-sample flux coupled with a more stable beam position enhancing existing capabilities. Furthermore, the upgrade will enable highly efficient operations (even prior to upgrade, the old APS ring operated at 99% beam delivery efficiency). The resulting impact will be noticeable across all platforms, including higher accuracy and precision of structural and pressure-volume-temperature measurements, improved signal-to-noise gain for inelastic/spectroscopy studies, expanded spatial resolution from ~100 nm to mm scale, and increased time resolution approaching single bunch MHz measurements, to name a few.

Accepting the new lower emittance APS-U beam and taking full advantage of the new x-ray source requires significant improvements to all beamline components used in delivery of x-rays from the storage ring to sector 16's experimental stations as well as enhancing the experimental capabilities. With nine total stations along the beam path, which are connected to the storage ring via four beam flight vacuum tubes and ranging from ~30 to 70 m distance from the source, the upgrade effort for the 20+ year old HP-CAT is substantial in scope, cost, planning, and execution. Following a comprehensive assessment in 2019, the upgrade plan for HP-CAT was initiated and full work scope plan was presented to HP-CAT's Executive Council (comprised of CAT partners, funding agency, and APS/APS-U management) in late-2019. Currently, HP-CAT is undergoing a full upgrade, which includes replacement and/or refurbishment of undulator insertion devices, monochromators, focusing optics, new stability-improved experimental tables, and a comprehensive upgrade to controls and various beamline equipment safety monitoring systems. With an estimated total cost of ~$14M, the HP-CAT upgrade is planned to proceed in phased approach across our beamlines. The overall footprint, with five experimental and four optics stations, and off-line laboratory space, will remain the same, but the vital infrastructure in beam delivery and experimental configuration will be significantly advanced. The phased upgrade approach is guided by the scope of effort pertaining to each station, the relative complexity of components and required timelines for detailed assessments/design/procurement/etc., and coordination with funding agency. HP-CAT's upgrade commenced even prior to start of APS-U and the overall phased upgrade effort is expected to overlap with APS-U downtime (~Apr. 2023 to ~Apr. 2024) and completed over a two-year period. Overall, the HP-CAT upgrade will result in substantial enhancement across all experimental platforms and an opportunity for implementation of new featured capabilities. In particular, and in addition to enhancement of all diffraction, imaging, inelastic, and spectroscopy techniques, the featured capabilities are based on optimizing the spatial, temporal, and x-ray coherence properties enabled by the new APS-U storage ring lattice. The upgraded state-of-the art APS and HP-CAT is well positioned to continue to drive the frontiers of extreme conditions research and serve the extensive array of our user community ambitions.

The HPCAT Workshop-2023 report was prepared by the workshop organizing committee, session organizers (as noted in breakout session sections of the report), and with contribution from all workshop participants. We are grateful for the continued contribution from the HP-CAT community, and we extend our deepest gratitude to all workshop participants, keynote speakers, and numerous contributors to this report.

-Nenad Velisavljevic HP-CAT Director and the HP-CAT team
Maddury Somayazulu (HPCAT Group Leader), Guoyin Shen (Principal Scientist), Arunkumar Bommannavar, Paul Chow, Tyler Eastmond, Innocent Ezenwa, Rich Ferry, Rostislav Hrubiak, Freda Humble, Curtis Kenney-Benson, Changyong Park, Dmitry Popov, Eric Rod, Dean Smith, Jesse Smith, and Yuming Xiao

Participants of the workshop photographed with Director Kimberly Budil of LLNL and Director Paul Kearns of ANL.

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Introduction to HP-CAT

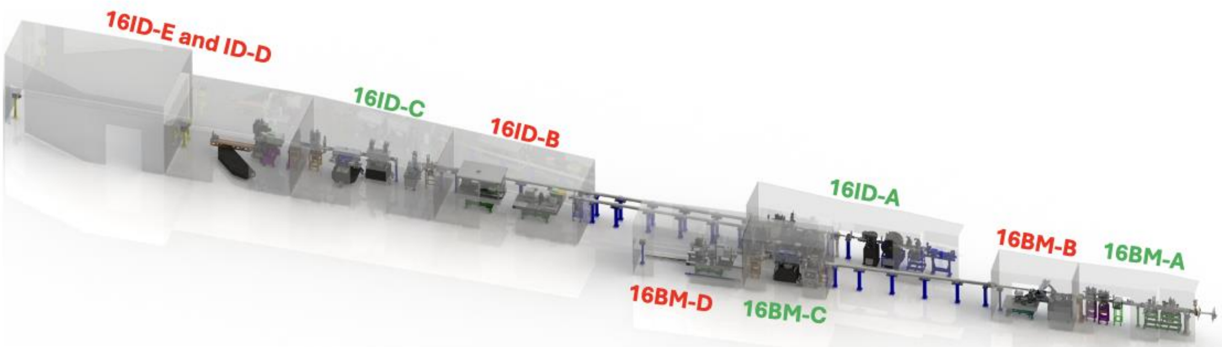
The High Pressure Collaborative Access Team (HP-CAT) was established as a partner facility (i.e. CAT model). From the beginning, the DOE National Nuclear Security Administration (NNSA) Office of Experimental Sciences has been the majority sponsor of HP-CAT. To improve overall management structure and operations of the facility, in 2018 NNSA established a management agreement and partnership model. HP-CAT is currently soley funded by NNSA Office of Experimental Sciences and the core partnership group includes research groups funded by Science Campaign 2 at the three NNSA Laboratories (Lawrence Livermore National Laboratory, LLNL; Los Alamos National Laboratory, LANL; and Sandia National Laboratories, SNL) and the NNSA Stewardship Science Academic Alliances (SSAA) program grant recipients. In addition to core partner group, which receive guaranteed 75% of experimental beamtime, HP-CAT also allocates 25% of beamtime to General User (GU) groups. The GU program is coordinated directly by APS and proposals submitted from PIs around the world are evaluated independantly by the APS Proposal Review Panel (PRP) which consists of representatives from a broad range of external institutions and subject matter experts in the field - https://www.aps.anl.gov/About/Committees/Proposal-Review-Panels. In addition to the experimental time, HP-CAT also allocates ~10% of available beamtime to research and development (R&D) of new cutting-edge x-ray capabilities and high-pressure apparatus. R&D beamtime provides a great opportunity for engagement between HP-CAT, partner groups, and the GU community, in working together to drive advancements in extreme conditions research and ultimately fostering the mutually benefical evolution of the facility.

HP-CAT is a dedicated facility for high pressure (HP) research with application of Diamond Anvil Cell (DAC) and Large Volume Press (LVP) type platforms, as well as introduction of next generation apparatus for generating extreme conditions. HP-CAT personnel located at the APS are committed to operating and maintining all beamlines, which consist of a Bending Magnet (BM) branch and an Insertion Device (ID) branch. In total, HP-CAT currently consists of nine stations (five experimental and four optics stations), as well as various off-line support laboratories.

Prior and up to the start of the APS-Upgrade (APS-U), HP-CAT operated five experimental stations, with four operating simultaneously. Overall, HP-CAT includes a bending magnet (BM) branch and an Insertion Device (ID) branch. The BM branch consisted of BM-B and BM-D experimental stations operating simultaneously in parallel. The ID branch includes ID-B, ID-D, and ID-E experimental stations. With a canted undulator setup, ID-B and ID-D/E operate independently in parallel (with ID-D and ID-E sharing the same undulator and working in series only).

- **16BM-B**. Multi-array capabilities, including Paris-Edinburgh (PE) type LVP and white-beam Laue x-ray microscopy. The PE press, which can generate up to ~8GPa and ~2500K, can be used to obtain in situ structure and viscosity measurements of melts/liquids, longitudinal and shear elastic moduli from a coupled piezo-transducer ultrasonic setup, and radiographic imaging over the range of pressure-temperature conditions. The white-beam Laue technique is tailored for measurements with DAC and provides cutting-edge measurements of structural deformation and transformation mechanism of materials during high-pressure loading.

- **16BM-D**. Dedicated x-ray diffraction for DAC measurements. In addition, x-ray absorption spectroscopy (XANES and XAFS) can be performed in tandem with diffraction in 16BM-D, which is a unique capability that helps unravel microscopic mechanism of pressure-induced changes.
- **16ID-B.** Microdiffraction beamline, with a general purpose (GP) and a dedicated laser heating table (LH) setup, that can be used for studies of a broad range of materials and pressure-temperature conditions.
- **16ID-D and ID-E**. Dedicated x-ray spectroscopy and inelastic x-ray scattering with DAC. Main capabilities include XES, XRS, NFS, and NRIXS. Furthermore, with joint ID-D and E setup, these stations have been used for specialized setup test measurements, including time-resolved XRD, SAXS with DAC, and other complex measurements.



Schematic layout of HP-CAT sector 16 at APS. The red and green font labels indicate the five experimental stations and four optics stations, respectively.

In line with APS-U, HP-CAT has made significant investments in all capabilities and especially tailored toward optimizing use of enhanced brightness and coherence. Likewise, various layout modifications had to be made due to the impact from APS-U – especially on our BM branch. Introduction of the new MBA lattice resulted in APS-U BM source shifting 1.826m upstream and 42.4 mm inboard (per APS surveying validation and measurements) relative to pre-upgrade storage ring setup. This resulted in a number of significant impacts, including: only M3 of APS-U BM source can be used (the M4 Dipole and reverse-bend Q8 quadrupole source is not adequate due to intensity-energy fluctuations); BM layout had to be completely redesigned and shifted to accommodate beam from new BM source; and ONLY part of radiation fan can be used (smaller radiation fan and shifted inward compared to pre-upgrade) and the loss of branching mode between BM-B and BM-D (i.e. post APS-U BM-B and BM-D cannot be operated simultaneously in parallel). Going forward, our new setup will still include five experimental stations, but only three working simultaneously (compared to four pre-APS-U).

- **16BM-B**. Dedicated station for Paris-Edinburgh (PE) LVP experiments. The PE press, which can generate up to ~8GPa and ~2500K, can be used to obtain in situ structure of melts/liquids using EDXD, viscosity measurements of melts/liquids, longitudinal and shear elastic moduli from a coupled piezo-transducer ultrasonic setup, and radiographic imaging over the range of pressure-temperature conditions.

- **16BM-D**. Dedicated station for DAC XRD and white-beam Laue technique measurements.
- **16ID-B.** Microdiffraction beamline, with a general purpose (GP) and a dedicated laser heating table (LH) setup, that can be used for studies of broad range of materials and pressure-temperature conditions from ambient to >500 GPa and ~10K up to 4000+K.
- **16ID-D and ID-E**. Dedicated station for XES, XRS, NFS, and NRIXS. Note, the new APS-U bunch mode will impact NFS and NRIXS measurements and limit range of materials that can be investigated. Future plans may include fielding XANES and EXAFS as well, which was previously available at 16BM-D. Furthermore, the plan is to also focus on optimizing the ID-E station and introduce new capabilities for sub-micron beam XDI, BCDI, time-resolved XRD, and PCI measurements with DAC.

Major changes from pre-upgrade to post APS-U

- HP-CAT will retain five experimental stations, but only three working simultaneously (compared to four pre-upgrade).
- BM branch fully restructured, due to a shift in BM source with new MBA lattice.
- Loss of branching mode with BM-B and BM-D experimental stations. *Pre-upgrade* BM-B and BM-D operated simultaneously in parallel and *post-upgrade* BM-B and BM-D will work in series ONLY.
- BM-B and BM-D experimental capabilities restructured and going forward BM-B will be dedicated to LVP experiments and BM-D dedicated to DAC measurements.
- ID-B will retain its exceptional capabilities with a new revolver undulator, new monochromator, new focusing optics, new detectors and new experimental tables for maximixing stability.
- ID-D and ID-E will remain dedicated for spectroscopy and inelastic x-ray scattering. We plan to optimize use of ID-E station and introduce new capabilities, including sub-micron beam XDI, BCDI, time-resolved XRD, and PCI measurements with DAC. The two stations will then continue to operate in series, but with broader range of experimental capabilities.

HP-CAT will remain focused on delivering cutting-edge x-ray capabilities and HP platforms that are tailored for studies of matter under extreme pressure conditions.

Introduction to HP-CAT Upgrade

The strength and success of HP-CAT operations is in the ability to develop a variety of x-ray probes (scattering, imaging, spectroscopy) optimized for Diamond Anvil Cell (DAC) and Large Volume Press (LVP) type platforms and being adaptable to the introduction of novel apparatii for generating extreme conditions. This has not only resulted in many scientific discoveries but also answered several deeper questions regarding the nature of physical phenomena at extreme pressure-temperature conditions. This adaptable approach will remain as HP-CAT's guiding philosophy moving forward, with an added goal of fully utilizing the higher brightness, enhanced coherence and finer focus with the new APS-U beam. Leveraging these new capabilities presents challenges. For example, although leveraging improved beam coherence has been demonstrated in pioneering studies (Le Bolloc'h, Itié et al. 2009, Yang, Huang et al. 2013, Husband, Hagemann et al. 2022), its full potential hasn't been more systematically demonstrated for DAC experiments. APS-U's high coherence above 20 keV provides the opportunity to further optimize use of beam coherence and apply it to a broader range of high-pressure measurements. Another aspect of APS-U is the timing pattern, which will include a 48 and 324 bunch mode (previous hybrid mode no longer available). This may present some challenges for single-shot experiments. HP-CAT is, however, committed to finding innovative solutions for reliable time-resolved experiments to fully exploit the brightness and single-photon counting detectors for shutter-less data acquisition (Fröjdh, Bergamaschi et al. 2024).

The HP-CAT-Upgrade project will align with the APS-U and install a new set of beamline components to (1) optimize x-ray beam brightness in a wide energy range for various x-ray techniques, (2) preserve the enhanced coherence at end experimental stations, (3) maximize the on-sample flux for time-resolved and efficient scanning (or mapping) measurements, and (4) achieve the best beam-pointing stability. Meanwhile, instruments in the experimental stations are under major upgrade, including advanced focusing optics, state-of-the-art controls for high-pressure devices, and simulated designs that maximize the stabilities in both position and environment (such as temperature). HP-CAT-U will be completed in several phases. In the first phase, the commissioning of 16ID-B and 16BM-B is planned to be able to bring them on-line. The general purpose (GP) table and laser heating (LH) table in 16ID-B will enable micro-diffraction with a refurbished branching monochromator that selects the beam from a revolver 21/25 undulator. This highly stabilized beam will be focused by new KB mirrors to a 2 μm^2 beam that has higher flux and better profile to enable high pressure-temperature experiments with ramped compression and/or ramped heating. The 2 μm^2 beam size is chosen based on extensive experience in DAC experiments in the past 20+ years, meeting the majority needs of users' experiments. The commissioning of 16BM-B with a focused white beam and new

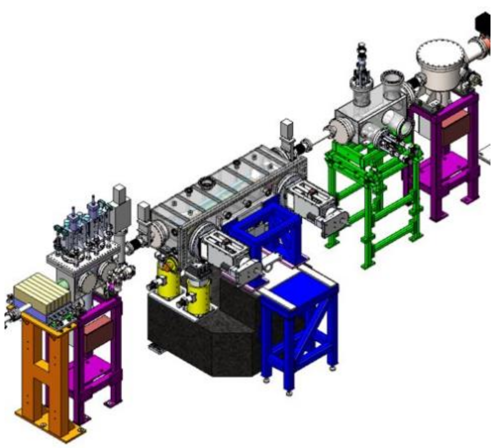


Figure 1: Rendering of the components in 16BM-A post upgrade. The figure above shows the 1M long KB mirror repurposed from 16ID-C which will be used to condense the horizontal beam.

experimental table and detector optimizes the x-ray techniques using the Paris Edinburgh Press (PEP) – LVP type device available at HP-CAT – program to the wider user community. Concurrently, while 16BM-B is being commissioned, we will commission the high-throughput double multilayer monochromator in 16BM-C (now configured to deflect in the horizontal geometry). The commissioning of 16BM-D will include a new granite-based experimental table, new focusing mirrors, and new detectors, all optimized for micro-diffraction using monochromatic beam (ΔE of 1-2 eV), pink beam (ΔE ~ 100 eV), or white beam. In the second phase, we will be commissioning our newly procured horizontally deflecting double-crystal monochromator (HDCM) and the horizontally deflecting multilayer monochromator (HDMM) in 16ID-C which will enable the spectroscopy station in 16ID-D. These new monochromators, designed to match the new revolver undulator and APS-U beam will significantly enhance the x-ray spectroscopy techniques of 16ID-D. With the incorporation of the pre-figured mirrors in 16BM-D and the Compound Refractive Lenses (CRLs) in 16ID-C in phase three, sub-micron diffraction and imaging experiments become possible. The major component in phase four is the commissioning of the 16ID-E station. The detail upgrade scope is currently in progress and contingent on availability of future funding investment. Timelines for resumption will proceed following completion and resumption of ID-D station

Our high-throughput endstation, 16ID-B will continue to support micro-diffraction at extreme pressure-temperature conditions. Furthermore, the feasibility of sub-micron focus possible in 16ID-E will extend the capabilities of HP-CAT to enable X-ray Diffraction Microscopy (XDM) and introduce advanced Bragg Coherent Diffraction Imaging (BCDI) and Phase Contrast Imaging (PCI) techniques. With the installation of a HDMM in 16ID-C, the increased brightness will similarly enable photon-hungry experiments both in 16ID-D (for time resolved XES, for example) and 16ID-E (for TPa diffraction and time-resolved diffraction measurements, for example). Similarly, the inclusion of a long (1M) focusing condenser mirror in 16BM-A enables PDF measurements of amorphous materials in a double-stage PEC over a broader pressure regime. Incorporation of complimentary multilayer optics in 16BM-B will facilitate phase contrast imaging of mm-sized samples at high P-T conditions. The increase in flux possible with such condensing optics, along with better detectors, is expected to expedite data collection for PEC experiments and offset some of the beamtime lost due to the tandem operational model of the BM line at HP-CAT. Incorporation of complimentary multilayer optics in 16BM-B will facilitate phase contrast imaging of mm-sized samples at high P-T conditions. Furthermore, the commissioning of a dedicated instrument for white beam, pink beam, and monochromatic diffraction in 16BM-D, together with the sub-micron focusing optics, will provide the much-needed boost for understanding the role of defects and non-hydrostatic stresses in behavior of materials at extremes of pressure and temperature. Some of the instrumental enhancements and newer experimental capabilities are summarized below.

Maximizing brightness, coherence, and on-sample flux: At HP-CAT, varieties of x-ray techniques, such as spectroscopy, diffraction, and imaging, require different energies of the incident photons for optimizing S/N ratio, detector efficiencies, DAC geometry and timing structure. To leverage the best of all these factors from the upgraded storage ring, a decision was taken to opt for two revolver undulators (the current HP-CAT front-end is canted and utilizes one undulator for ID-B side-branch and one for the ID-D-E main branch) that can either enhance brightness in the smaller energy ranges 10-15 keV, 25-35 keV or offer continuous tunability between 10-65 keV with enhanced brightness at high energies (>50 keV). In addition, to preserve coherence, the canted beamline 16ID-A-C-D-E will have a windowless design, with its first window at the entrance point of the 16ID-D experimental station. Further enhancement of brightness will be achieved by introduction of HDMM in tandem with HDCM. The HDMM will benefit from improved symmetrical harmonics of the APS-U undulators and will allow a wide bandpass (~1% in the 20-30 keV range) beam which would deliver an additional 1-2 order of magnitude increase in flux for performing time-resolved experiments.

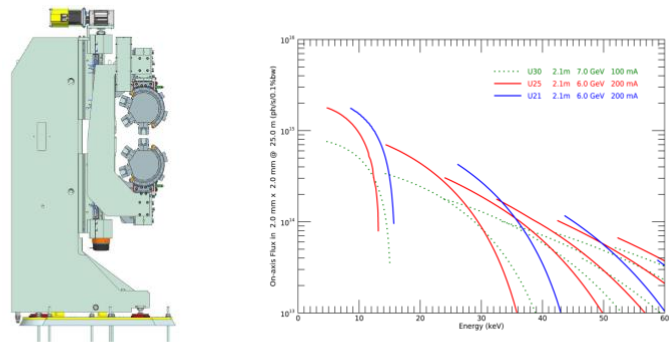


Figure 2: Schematic of a revolver undulator is shown on the left with three magnet assemblies while the plot on the right shows the on-axis brightness curves for the two periods (2.1 and 2.3) for the HP-CAT revolver undulators.

X-ray beam stability: Stability of the incident beam is critical in the quest for sub-micron focusing and important for shutterless, time-resolved measurements. This dictated the need to modify the existing branching monochromator that services 16ID-B. The LN_2 cooled first crystal assembly of the branching monochromator (BDCM1) that houses three crystals (Si220, Si311 and Ge111) has shown structural instability causing intensity excursions at the sample tables. The performance of the monochromator in view of the higher local heat loads after the APS-U was also an additional consideration in the design and refurbishment of the monochromator. Additionally, to minimize the effect of a long lever arm that results from having the High energy Double Crystal Monochromator housed in 16ID-A pre-upgrade, the optics station components serving 16ID-B and 16ID-C-D will be fully decoupled. This change was also possible since HP-CAT can accept the undulator white beam in 16ID-C and the smaller emittance of the APS-U beam obviates the need for the long (1.2 m) KB mirrors that serviced the spectroscopy experiments in the past. This also allows for a windowless setup and intercepting the beam with a single diamond window after 16ID-C, thereby maintaining the highest beam coherence for experiments in 16ID-D/E. Further, the environmental stability and minimizing the effect of temperature drifts on the experimental tables is expected to be the limiting factor in minimizing the drift of the sample from a focused incident beam. This has been addressed by investing in a highly stabilized HVAC system with an overall temperature stability to within ±0.1 C inside the end-stations.

Precision instrumentation for end-stations at HP-CAT: The experimental tables are all being replaced by specially designed granite tables that allow better access to sample stages. The sample stages are also being replaced by air-bearing stages that minimize mechanical imprecision. The focusing mirrors are being replaced with state-of-the-art mirrors that have superior figures of merit and are expected to not only offer better focus, but also better beam profiles. Simulations carried out by the XSD optics group indicate that the limiting factor would be the bender errors rather than the mirror surface error. Two new detectors with CdTe sensors (PILATUS3 2M and 1M from DECTRIS) have been purchased and commissioned in 16ID-B and 16BM-D, respectively. With the pre-upgrade beam, we have observed an order of magnitude increase in detection efficiency allowing us to perform mSec exposures on ramp laser heated samples (Kim, Oka et al. 2022) or in dynamically compressed samples with the dynamic DAC (Evans, Yoo et al. 2007) (Biwer, Quan et al. 2021, Copley, Smith et al. 2023).

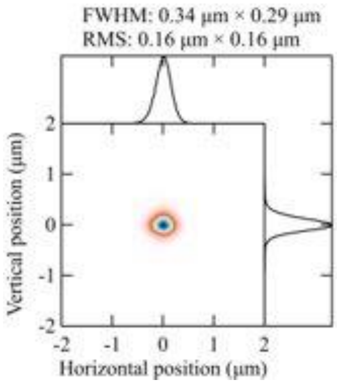


Figure 3: Simulated profile of the focal spot from a 200 µm incident beam at 30 keV from a U25 undulator on the GP table in 16ID-B.

Bending magnet beamline: The change in the magnetic lattice affects HP-CAT in the utilization of the bending magnet line and in variance with how we have been operating to date. Post APS-U, 16BM-B with the Paris Edinburgh Press (PEP) will operate in tandem with the 16BM-D Laue diffraction table. To enhance the capabilities of both these hutches, we are deploying a 1.2 M long KB mirror in 16BM-A that can condense a 1 mm horizontal white beam into a focal spot of roughly 20 µm at the sample position and enhancing resulting flux by roughly x7. Similarly, we are investing in a pair of pre-figured mirrors from JTec that can focus a 150x150 µm white or monochromatic (or wide band-pass polychromatic) beam to a sub-micron spot on a versatile diffractometer specifically designed for DAC experiments (shown in the figure alongside) in 16BM-D. The PEP experimental table has also been redesigned for combining diffraction, tomography, radiography, and includes a vertically deflecting multilayer that can enable phase contrast imaging (Kono, Kenney-Benson et al. 2015). A hexapod and a rotating coupling (of the pressure-transmitting, hydraulic fluid pipe) enables precision rotation of the PEC that will open computed tomography of amorphous or crystalline samples in a PEC (Philippe, Le Godec et al. 2016, Ohta, Wakamatsu et al. 2020). To complement these upgrades, a state-of-the-art Ge detector has been purchased that allows faster data measurements with smaller dead time and larger dynamic range. Overall, these improvements will not only enable faster measurements but will also contribute to rapid data acquisition, especially for PDF measurements needing to cover a larger q range by collecting data at multiple detector angles (Eastmond, Hu et al. 2023).

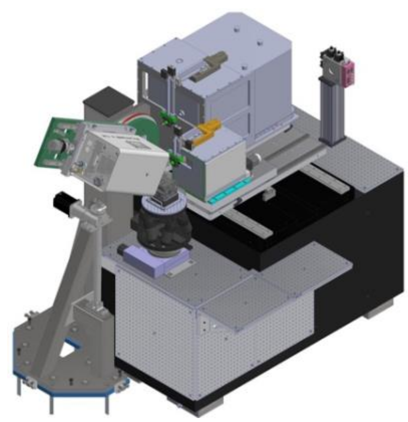


Figure 4: Newly designed experimental table in 16BM-D

New monochromators that enhance existing capabilities and enable new techniques: The HDCM shown alongside is equipped with Si111 and Si311 crystals in a horizontal rotation that will allow higher reproducibility and stability of the monochromatic beam. Stationed in 16ID-C, it will service both the spectroscopy needs in the 4.5 – 20 keV energy range and high energy needs of the coherent diffraction and imaging experiments in the 16ID-E hutch at 20 – 50 keV energy range. Coupled with the double multilayer monochromator (HDMM), we will have the option of using either of these monochromators to be able to perform ‘pink’ beam experiments with higher flux using the HDMM, or carry out monochromatic beam experiments with high resolution including XES, XRD, and PCI. To utilize the larger demagnification ratio afforded at 16ID-E, which is roughly 70 M from the source, double focusing with a train of CRL lenses in 16ID-C and table-top prefigured KB mirrors is expected to offer sub-micron focal spot that will enable some of the photon-hungry toroidal anvil (tDAC) experiments as well as dDAC higher strain rate studies. The diamond CRL ‘transfocator’ (Xray , Narikovich, Polikarpov et al. 2019) planned in 16ID-C will be able to focus the 5-25 keV x-rays to the sample stack in 16ID-D located 5 m downstream and enable SAXS experiment in 16ID-D/E. In the future, this transfocator could also be used to enable energy-dispersive XES and XAFS studies using the HDMM beam and a von Hamos analyzer (Alonso-Mori, Kern et al. 2012, Pascarelli, McMahon et al. 2023, Sahle, Gerbon et al. 2023).

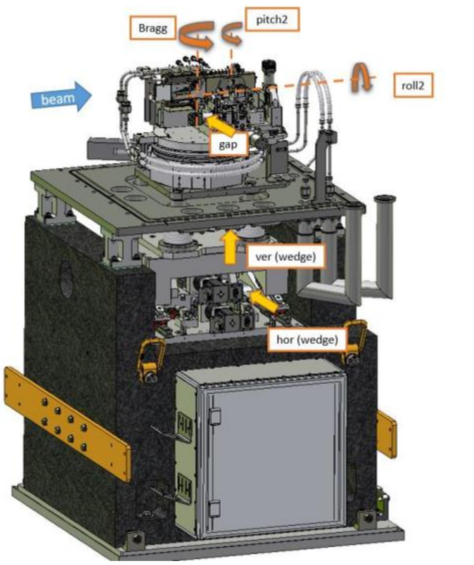


Figure 5: HDCM that will be installed in 16ID-C

Upgrade status and path forward: Overall, HP-CAT has undergone a significant upgrade encompassing all layout/instrumentation and enhancements, which has been planned and implemented in a phased approach. With ~$10M invested in key infrastructure so far, we are planning to compete for the remainder of the estimated $14M total cost and ultimately complete resumption across all nine stations (five experimental and four optics stations). HPCAT-U commenced concurrently with the start of APS-U shutdown (Apr. 2023 – May 2024) and is planned to be completed in the next two years, as funding and major equipment is installed and commissioned.

With the completion of this workshop report (August 2024), following a yearlong shut down for upgrade, APS resumed operations by first delivering beam to five core APS operated beamlines, and on July 03, 2024, HP-CAT received its first x-ray beam post-upgrade in the 16-ID-A station. It took significant effort to be first in line, following the opening of several core APS-operated beamlines, to receive x-rays and HP-CAT will push toward resumption and user support in a phased approach that is based on upgrade progress, funding availability, and scheduling with APS Commissioning Readiness Review Team (CRRT).

Commissioning status and user resumption schedule:
Phase 1 – 16ID-A-B Stations

- Currently, following first light received on July 03, 2024, ID-B station is undergoing commissioning to optimize the beam and provide x-ray specs to users (which is critical toward accepting users/GUPs)
- We expect ID-B to be ready for users by the later part of 2024-3, at which time HP-CAT will introduce users via Rapid Access Proposals (RAP). At the same time, we plan to open the GUP proposals for ID-B for the 2025-1 cycle

Phase 2 – 16BM Branch

- BM branch, as noted in section 4, had to be completely restructured to deal with APS-U impact
- Currently, BM has been redesigned (with subcontract to IDT vendor) and components for the new setup are arriving and expected to be completed by ~Oct. 2024
- BM branch is scheduled to receive first light ~late 2024-3 cycle and we will subsequently work on commissioning, followed by first users via RAP in 2025-1 and GUPs for 2025-2

Phase 3 – 16ID-C-D-E Stations

- All three station (C, D, and E) have undergone a full assessment and major configurational changes
- New HDCM and HDMM have been ordered (Axilon industries) and expected to be received ~late 2025-1 cycle for install in ID-C and subsequent performance evaluation and optimization
- ID-D is expected to get beam back in 2025-2 and proceed with commissioning toward user access in 2025-2-3 cycles
- The ID-E station upgrade is currently in progress and contingent on availability of future funding investment. Timelines for resumption will proceed following completion and resumption of ID-D station

HP-CAT-2023 Invited Presentations (Highlights)

The first session of the workshop was focused on celebrating the two decades of operations of HP-CAT and the excellent science that it resulted in. LLNL Director Dr. Budil and ANL Director Dr. Kearns highlighted some of the major achievements, and especially noting the synergetic aspects of HP-CAT among partners, user community, and Department of Energy, in helping foster a highly productive scientific community and growing HP-CAT into the largest synchrotron-based high-pressure research facility in the world. As noted by HP-CAT and APS directors, and various speakers throughout the workshop, the vision of the founding members in 1999 to create a cutting-edge user facility and attract a diverse community for advancing research in extreme conditions has not only been realized but has also surpassed expectations.

Opening the scientific discussion, both the current APS Director L. Chapon and former APS Director B. Stephenson highlighted the exciting opportunities with the increased brightness and coherence of the post APS-U beam. Both speakers also noted that taking full advantage of the MBA lattice and enhanced coherence and higher brightness will require continued, and even stronger, synergetic collaboration of the community. Many of the much-awaited scientific discoveries can be achieved, especially in imaging, ptychography and high energy x-ray microscopy, but this will also require *complementary* AI/ML tools in data analysis that are critical in decoding the high-volume data. As an example, Dr. Stephenson further amplified this point in highlighting the scientific opportunities that new methods such as pink beam powder diffraction, surface microdiffraction, X-ray photon correlation spectroscopy (XPCS), and Bragg coherent diffraction imaging (BCDI) will make possible. These techniques directly benefit from higher coherence, higher brightness and cleaner undulator spectra. The near-atomic-scale beam focusing (~1 nm^2 focused beam from Laue lenses) will enable probing atom level dynamics, whether in situ on deposition layers of CVD systems or nanoscale strain from BCDI measurements. These techniques would allow probing the local strain in micron sized samples at TPa pressures and provide unprecedented insight into material at extreme conditions.

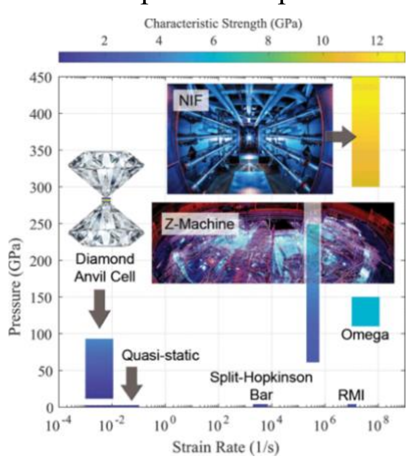


Figure 6: Characteristic conditions of pressures and strain rates for various HPHT techniques. The workshop featured talks from experts of all the above platforms. Figure reproduced from (Brown, Adams et al. 2019).

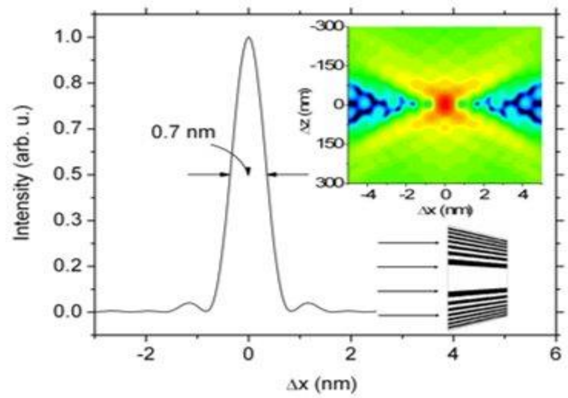


Figure 7: Simulations predict near atomic scale focusing with a wedged multilayer Laue lens. Based on data from (Yan, Maser et al. 2007)

HP-CAT, as a facility also represents a very large high-pressure research community, and the keynote speakers included representatives covering a broad range of our user groups and scientific interests/disciplines. In some of the talks, representatives from NNSA laboratories (LLNL, LANL, and SNL) focused on the opportunity to further advance x-ray capabilities for investigating material behavior beyond crystal structure and P-V-T measurements and gain deeper insight into kinetics, transformation, and deformation mechanism during compression. For example, recent work which leveraged complementary radial-diffraction in DAC, ultrasonic sound speed measurements with PEP, and various laser/gun dynamic measurements, have provided the first in-depth assessment of material strength of Sn and Ta. With the new beam and x-ray techniques, new measurements, such as White-beam Laue and BCDI, could be applied to further support the strength models and develop mesoscale modeling over extended length and time scales. In a number of specific examples, it was noted that while higher strain rates (>10^5/s) tend to show larger deviations where the strengths of materials are not well characterized, these measurements allow us to infer strength better than extrapolating from smaller strain rates (<10^3/s). Understanding the coupled effects of strain rates, phase transitions, pressure and temperature requires developing better theoretical frameworks and data from several platforms, which seems to amplify the role of dislocation self-multiplication.

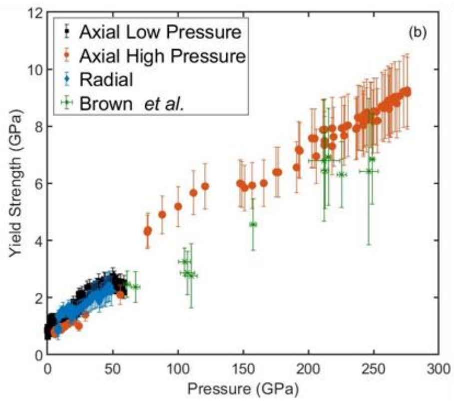


Figure 8: Results of the radial diffraction studies on Ta from measurements performed at 16BM-D (Brown, Adams et al. 2019).

Experiments that understand material behavior across several rates, especially at lower pressures (<200 GPa) and combined with grain resolved diffraction, which becomes possible at HP-CAT with the APS-U beam, are key to validating such simulations. Overall, the push to bridge strain rate gap (region between standard static DAC measurement, to quasistatic up to 10^3/s strain rate d-DAC measurements, and all the way up to 10^7/s strain rate laser/gun/pulsed-power measurement) will be vital in establishing comprehensive modeling/theory framework and gaining clearer understanding between fundamental measurements (as done with x-ray) and bulk functional material properties.

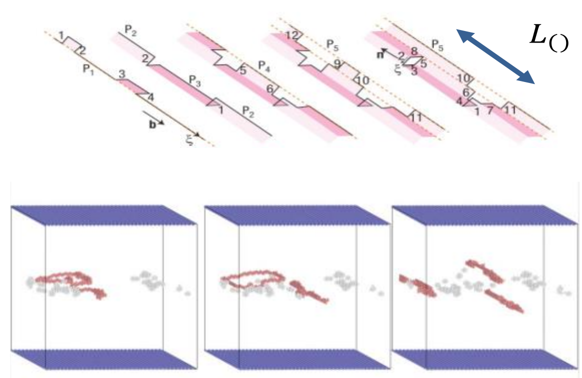


Figure 9: Results of atomistic simulations reveal cross-linking of dislocations (top figure) as well as self-multiplication of the dislocations as one possible mechanism for the abrupt increase of hardness at high strain rates. Based on data from (Zepeda-Ruiz, Stukowski et al. 2021).

Representatives from national laboratories, academia, and other institutions also raised the challenges posed by the substantial advancements and introduction of new advanced materials. From High Entropy Alloys (HEAs) to additively manufactured and quantum materials, these new materials require robust and rapid testing platforms. R&D of new materials, although heavily guided by theoretical and modeling efforts, can involve trial and error and require testing at high P, T, and strain rates as a part of the production improvement feedback loop. The community overall continues to push the boundaries of discovery, and although the researchers come from a range of scientific disciplines, the common voice echoed that x-ray platforms and new material discoveries can only evolve in tandem.

One of the key parts of the workshop also focused on the evolution of x-ray facilities around the world (including synchrotron, XFEL, and in-house sources) and various large scale extreme conditions facilities (such as NIF, Z-Machine, Omega Laser facility, etc.). Several talks provided exciting updates, for example at EuXFEL and recent ICF achievements at NIF, which emphasized the collaborative role between these facilities and identified some gaps that could be addressed by HP-CAT and synchrotron-based facilities. Overall, there has been remarkable progress and achievement across all light sources and large-scale extreme conditions platforms. It is clear that each of the facilities provides some unique aspects, as well as some general overlap, but it is also clear that no facility can provide solutions to all problems. Rather, the connection and collaborative effort is vital to address the evolving landscape of extreme conditions research and to continue to push the boundaries in research.

High level summary of key points raised by invited speakers:

- Sustaining the synergy that helped HP-CAT grow over the first 20+ years will be even more critical going forward and ensuring continued success.
- APS-U and the new synchrotron beam provide an array of opportunities, but taking full advantage of higher brightness and coherence to develop cutting edge x-ray techniques requires sustained R&D and strong collaborative efforts.
- Scientific discoveries from cutting edge x-ray measurements will also require concurrent investments in high performance computing platforms, including AI/ML, to handle complex and high-volume data.
- Pushing to bridge strain rage gap (region between standard static DAC measurement, to quasistatic up to 10^3/s strain rate d-DAC measurements, and all the way up to 10^7/s strain rate laser/gun/pulsed-power measurement) is vital in establishing comprehensive modeling/theory framework and connecting fundamental measurements (as done with x-ray) and bulk functional material properties.
- Developing capabilities that not only focus on materials studied at HP-CAT now (e.g. metals/alloys, polymers, earth and planetary, strongly correlated, etc. materials), but being forward-looking and developing capabilities that will allow studies of advanced emerging materials (High Entropy Alloys (HEAs), additively manufactured, quantum materials, etc.).
- Need for further optimization of turnaround time for measurements – especially regarding the new-discovery materials – to accommodate faster proposal-to-experimental time process and avoid stifling discovery processes.
- Assessing the evolving landscape of light sources and large-scale extreme conditions platforms. Using the information to develop unique capabilities at HP-CAT, which

optimize synchrotron-based properties, while establishing integrated connection that allows for collaborative connection with all facilities to push the boundaries of the extreme conditions research field.

- Continue promoting HP-CAT as key connection for training students and providing pathway to national laboratory, academic, and other positions, as well as workforce sustainment at HP-CAT.

Report on Breakout Sessions – 1

Extending the Practical Limits of Pressure, Temperature and Strain rate

Earl O'Bannon, Zsolt Jenei and Daniel Sneed,
Physics Division, Lawrence Livermore National Laboratory
Vitali Prakapenka, and Dongzhou Zhang, SEES at Sector 13, University of Chicago
Dean Smith, University of Nevada at Las Vegas
Jesse Smith, HP-CAT-XSD, Argonne National Laboratory

The construction and upgrade of next generation synchrotron sources – together with the continual development of extreme-conditions apparatus and techniques – make it possible to explore the structure and properties of materials at ever-higher pressures, temperatures, and strain rates. The literature includes isolated examples of static pressure generation of up to 500 GPa or more, temperatures up to 5000 K or more, and strain rates as high as 10^3 s^{-1}. The Workshop's breakout session – *Extending the practical limits of pressure, temperature, and strain rate* – served as an exchange of information, techniques, challenges, and scientific opportunities related to experimental research at the very edge of what is currently possible using a variety of advanced diamond anvil cell techniques. The goal was two-fold: to make the current state-of-the-art more accessible to the broader extreme-conditions community, and to further extend the currently achievable limits.

There were three broad topics for discussion:

- Advanced apparatus for generating extreme pressures, temperatures, strain rates
- Best methods for measuring extreme pressures, temperatures, and strain rates
- Novel scientific opportunities at the limits of extreme conditions

While it is not possible to capture here the scope of the two-day discussions in its entirety, there were a few common themes or needs that emerged in the broader topics throughout the discussions:

- Improved source and beamline characteristics
- Advanced sample engineering and preparation
- Enhanced time-resolved capabilities
- Real-time visualization and post-experiment handling of large datasets

Improved source and beamline characteristics

Extending the limits of P, T, and strain rate requires the smallest, cleanest beam with the highest possible flux. A small, clean, focused beam is crucial because samples at ultrahigh pressures and temperatures have extremely steep radial pressure and temperature gradients. The high spatial resolution afforded by a small, clean beam will allow users to probe sample volumes at extremes while hopefully minimizing the contribution from lower-pressure and -temperature regions. The highest possible flux is needed because at ultrahigh pressure the sample is extremely thin, thus there is very little scattering volume. Furthermore, at ultrahigh temperatures and strain rates, it is necessary to collect useable diffraction data with the shortest possible exposure times.

The need for improved source and beam characteristics is being directly addressed by the APS and HP-CAT Upgrades. The APS upgrade, characterized by the multi-bend achromat lattice, will reduce the horizontal emittance of the electron beam down to the same order as the vertical emittance, resulting in a nearly circular, highly parallel source (Figure 10, center).

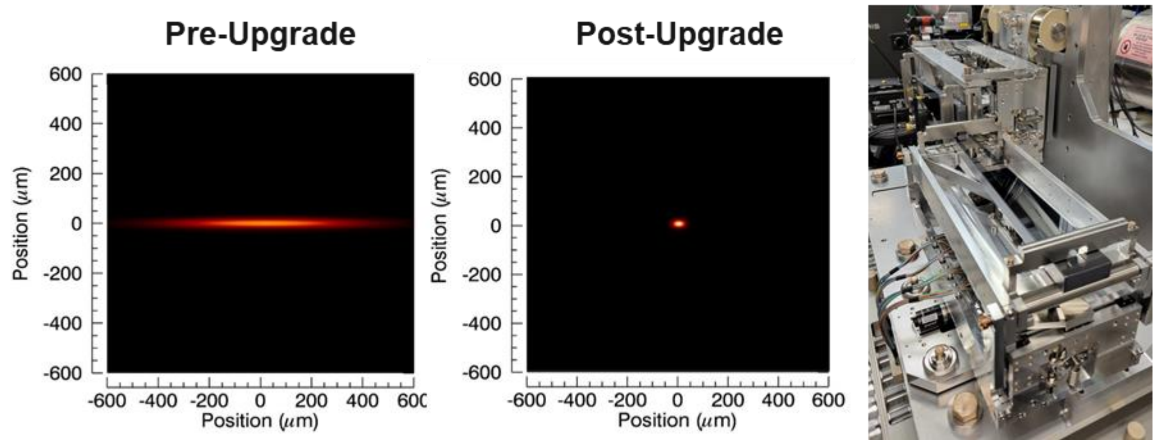


Figure 10**:** A comparison of the electron beam before (left) and after (center) the APS Upgrade. The small, approximately round source, coupled with the highest-quality reflective focusing optics (right), will provide HP-CAT users with very high flux of photons delivered in a small, clean, focused beam.

The HP-CAT Upgrade includes several pair of the highest quality reflective focusing (KB) mirrors, purchased from J-Tec (Figure 10, right). HP-CAT users should expect to experience a significant increase in flux (one to two orders of magnitude) while at the same time enjoying a smaller and cleaner focused beam (~2x2 μm^2 for large mirrors, better than 1x1 μm^2 for small mirrors) optimized for experiments at the very highest pressure, temperatures, and strain rates.

Advanced sample engineering

A well-engineered cell, a careful choice of seats and anvils, and meticulous sample preparation are all critical to reaching the most extreme pressure and temperature conditions, but even with the best tools and most careful preparation, there are practical limits to conventional DAC design and traditional loading techniques. A recent literature search by O'Bannon et al. (O'Bannon, Jenei et al. 2018) (Figure 11 left) reveals a practical limit of approximately 400 GPa for conventional anvils with single- or double-beveled culets. To push beyond this conventional limit, researchers have employed advanced anvil engineering techniques. Dubrovinsky, Dubrovinskaia, and coworkers employed a double-stage anvil assembly with nanocrystalline diamond hemispheres, claiming extreme pressures approaching (Dubrovinsky, Dubrovinskaia et al. 2012) and eventually surpassing (Dubrovinsky, Khandarkhaeva et al. 2022) 1 TPa. Multiple groups have used focused ion beam (FIB) milling to machine a so-called toroidal anvil (Dewaele, Loubeyre et al. 2018) (Jenei, O'Bannon et al. 2018). With this latter development, there are now several reports of pressures approaching, and even exceeding, 500 GPa. However, the sample size in this approach is still very small, on the order of 4-5 μm in diameter. The consensus at this Workshop was that the optimization of the toroidal design was the most likely and accessible path forward to routine ultrahigh pressures for the broader high-pressure community. Various toroidal features – including tip diameter, torus diameter, and tip height – can likely be tailored to suit the individual experimental requirements.

Reaching and maintaining ultrahigh temperatures requires great care during sample preparation. The thermal properties of diamond make it essential to have sufficient insulating layer(s) between the heated sample and the anvils. The laser heating community already understands this well, and there are many examples in the literature of methods and materials to insulate samples. The problem is magnified, however, at ultrahigh pressures as the entire sample chamber thickness is reduced to the order of microns. One strategy that was discussed during the workshop was to again turn to FIB milling to create shallow depressions on the culet faces – even just a couple of microns deep – to provide substantially thicker insulating layers at extreme pressures. One other approach would be to explore other toroidal anvil designs that provide larger sample volumes while maintaining the capability of generating pressures approaching 500 GPa. This, combined with advanced loading techniques such as micromanipulator-assisted loadings or even building complex sample packages using FIB techniques, could prove crucial in reaching the highest possible temperatures in a laser-heated DAC.

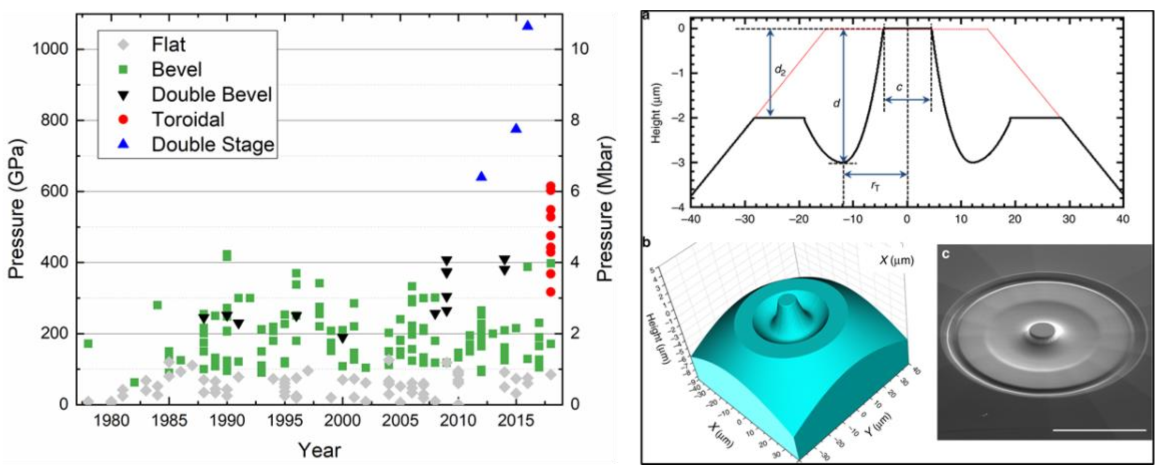


Figure 11**:** A survey of ultrahigh pressure experiments over the past few decades (left) suggests a practical pressure limit of about 400 GPa using conventional anvils. In the past decade there have been several reports of static pressures exceeding 400 GPa using double-stage and toroidal anvil designs. Modification of the relevant toroidal anvil design parameters (right) may be an important step in optimizing sample volume, sample chamber configuration, and ultimate achievable pressure. Left and right images adapted from (O'Bannon, Jenei et al. 2018, Perreault, Huston et al. 2022) (Jenei, O'Bannon et al. 2018). Reproduced from [O'Bannon et. al., Rev. Sci. Instrum., 89, 111501 (2018)], with the permission of AIP Publishing.

The highest measured strain rates using traditional static apparatus have been achieved using piezoelectrically driven DACs, or dDACs (Husband, Hagemann et al. 2022). The size and mounting requirements of the piezo stacks pose a challenge: on the one hand, one wants to make the mount as small as possible to facilitate the use of complementary equipment such as microscopes, pinholes, etc. On the other hand, the piezo has very limited stroke so the mount needs to be robust enough to sufficiently confine the piezo. Careful attention to these details, and the possible need to modify existing designs, such as using a lever arm mechanism, will be important moving forward.

Enhanced time-resolved capabilities

Enhanced time-resolved capabilities will be crucial to understanding time dependent processes and in advancing high strain rate and pulsed laser heating research. The mechanical performance and response of the dDAC is such that one can compress (at least thus far) faster than one can measure, and details are lost as the community is either flux- or frequency-limited. Figure 12 provides an excellent example, comparing two rapid compression experiments using the dDAC, each with a similar average compression rate of approximately 60 TPa/s. With the combination of increased flux available from the APS Upgrade and increased imaging frequency with latest-generation, large-format area detectors, the high-pressure community will be able to access the highest measured strain rates ever measured at a synchrotron source, with greater detail owing to improved time resolution.

Enhanced time resolution is critical for reaching ultrahigh temperatures as well. It is not feasible to maintain the very highest temperatures achievable in the laser-heated DAC for long time periods, as the insulating layers, themselves, can melt, reactions between and among the sample, insulating material, anvil, gasket, etc., can take place, and the sample chamber can become unstable resulting in diamond anvil failure. It becomes necessary to heat for very short periods of time. However, because the heat is initially applied to the surface of the sample, one does not want the heating to be too short, otherwise the sample may not be heated in its entirety (Goncharov, Prakapenka et al. 2010). Figure 13 shows there is an ideal pulse time, depending on the laser characteristics, sample volume, and sample properties, that will heat the entirety of the sample volume while minimizing the time that negative effects, such as melting, and reactions can affect the sample configuration. This sort of thermal modeling – together with precise timing and synchronization of heating, temperature measurement, and x-ray exposure – will prove crucial in reaching the highest possible temperatures in the laser-heated DAC.

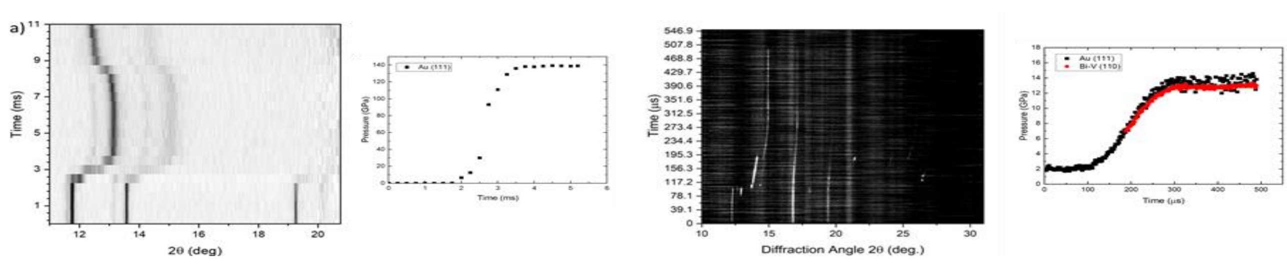


Figure 12**:** A comparison of the level of detail and information available from high-resoultion imaging. The composite image and corresponding plot on the left represent a compression rate of ~54 TPa/s, collected at a rate of 4 kHz (PETRA-III) while the composite image and corresponding plot on the right represent a compression rate of ~61 TPa/s, collected at a rate of 560 kHz (Eu-XFEL). The detail, density, and even oversampling of data has the potential to reveal minute or short-lived phenomena during and/or immediately following sample compression.

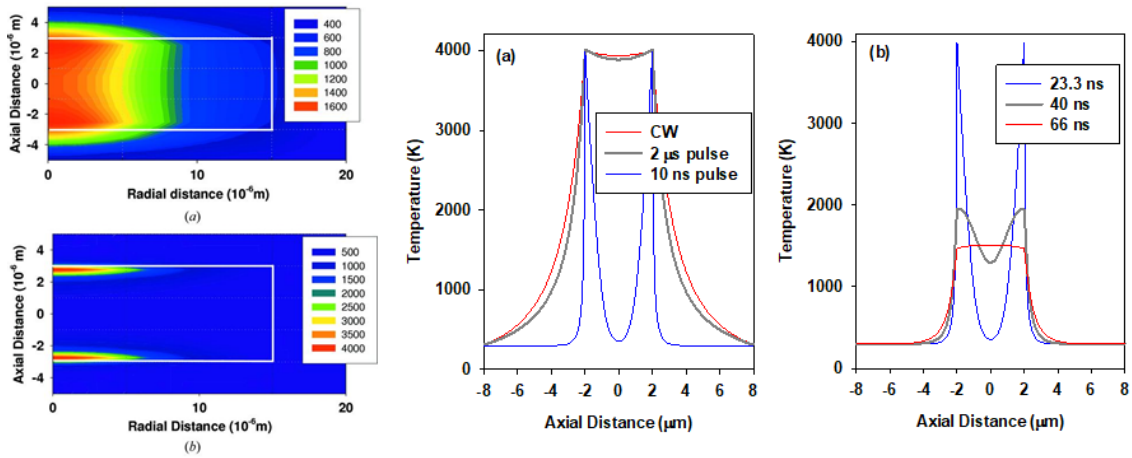


Figure 13**:** Modeling the axial heat flow and temperature distribution through a sample volume as a function of laser heating pulse duration suggests there is an optimum time range – in this case, on the order of a few tens of nanoseconds – to reach maximum temperature throughout the sample volume while minimizing some of the deleterious effects of melting and reactions that can result from continuous heating at very high temperatures in the laser heated DAC (Goncharov, Prakapenka et al. 2010). Based on data from (Goncharov, Prakapenka et al. 2010).

Real-time visualization and post-experiment handling of large datasets

Real-time visualization and evaluation of data at the beamline has long been an important aspect of high-pressure research. Samples are continually changing with changing pressure and temperature. Usually the changes are subtle, as a slight modification in pressure or temperature corresponds to a slight increase of decrease in lattice parameters, for example. But sometimes the changes are drastic, for example when the application of pressure and/or temperature drives a phase transition. Many of the needs associated with pushing the limits of pressure, temperature, and strain rate – increased flux, shorter exposure times, higher-frequency imaging – lead to progressively larger data sets. In some cases, it is no longer practical or even possible to carefully review all these data as they are collected. There is a clear need to develop strategies to handle large datasets.

One approach is to mix existing tools with new processing methods. For example, large datasets collected at relatively high frequencies could be processed using familiar tools such as Dioptas, but to do so rapidly and efficiently, the data could be moved to large servers where the images are processed in parallel, with composite images returned quickly to the user for precursory inspection. Often, these large datasets contain substantial amounts of redundant data, with just one or a few subset(s) of data of interest. These composite images could help identify one or more key points of interest for focused attention (or quickly reveal the dataset has not captured the desired phenomena). There is also the need for novel research tools. For example, AI or machine learning tools that can search a collection of images or other data for significant features that may be of interest could greatly reduce the time required to execute real-time data analysis and evaluation at the beamline. There is also a need to collect all the pertinent metadata associated with each experimental run. In a dDAC run, the experimentalist will need to know the parameters of the ramp, voltage applied to the piezo actuator, detector exposure time, and more, while for a high temperature laser heating run, much of the same information is needed along with laser power, pulse length, etc. This information should also be recorded in real time for each run. Finally, there needs to be efficient methods for data transfer following the experiment, and an understanding of the long-term storage and backup strategies by both the user and the facility.

Summary of challenges and opportunities

Extending the practical limits of pressure, temperature, and strain rate has the potential to reveal new phases, uncover novel material properties, help us better understand transition mechanisms, and advance extreme conditions research in ways we likely do not yet recognize. The improved source from the APS Upgrade, the improved hardware, software, and measurement techniques available at HP-CAT, and the innovative sample engineering and design from our members and the user community will make it possible to reach new limits in extreme conditions using a synchrotron source.

Report on Breakout Sessions – 2

Advanced Materials

Stella Chariton, SEES, University of Chicago
Yogesh Vohra, University of Alabama
Russell J Hemley, University of Illinois at Chicago
Innocent Ezenwa, Paul Chow and Maddury Somayazulu,
HP-CAT-XSD, Argonne National Laboratory

Material science has now come to recognize that the marriage of two fields – pressure-induced modification of bonding (and stoichiometry), and the success of crystal structure prediction with first-principles calculations – has resulted in a paradigm shift in our quest for designing new materials(Zhang, Wang et al. 2017, Miao, Sun et al. 2020, Xu, Li et al. 2022). This interplay has produced near-room-temperature superconductors (Flores-Livas, Boeri et al. 2020), superhard materials (Bykov, Chariton et al. 2019), electronic materials (Kim, Stefanoski et al. 2015, Wang, Hanzawa et al. 2017), and high energy-density materials (Eremets, Gavriliuk et al. 2004), to name a few. The challenge, however, remains in finding metastable synthesis pathways that can facilitate recovery of these materials upon pressure release to ambient. The other experimental challenge is the validation of these superior properties both at high pressures (and sometimes concomitant high-temperature conditions) and establishing the thermodynamic and kinetic release pathways that can maintain these properties (Amsler, Hegde et al. 2018, Dubrovinsky, Khandarkhaeva et al. 2022). In addition to synthesizing these materials from TPa pressures and temperatures above 2000 K (Walsh and Freedman 2018), there are synthesis pathways which explore high-pressure and concomitant shear (Gwalani, Olszta et al. 2020) (Santoro, Gorelli et al. 2013). This exploding field of mechanochemistry under high pressure has systematized the conventional ball-milling synthesis routes resulting in much better characterized and tailored sample synthesis strategies (Levitas 2004). Validating novel structural alloys, ceramics, and additively manufactured materials for their performance (or non-performance) at high pressure-temperature conditions or under non-hydrostatic loading has been hitherto limited by the availability of experimental probes that can interrogate a sample in a diamond anvil cell, in turn, restricting the P, T, σ conditions that can be brought to bear on the sample under question. One can also add to these other variables such as time and photon irradiation making the diamond anvil cell one of the most versatile sample platform for such studies (Boehler 2005) (Evans, Yoo et al. 2007) (Shen and Mao 2017) (Dong and Vayssieres 2018) (Lang, Zhang et al. 2010).

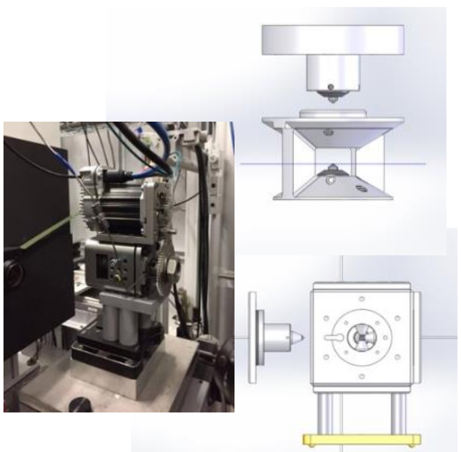


Figure 14: The RotoDAC, TomoDAC and MDAC are three variants of the cells designed at HP-CAT for leveraging APS-U and performing measurements that can highly benefit studies on discovering advanced materials and exploring the recovery pathways.

The breakout session on Advanced Materials was aimed at bringing together the material scientists that synthesize, validate, and predict both materials synthesized at extreme pressure-temperature-strain conditions as well as materials for extreme environments. The aim was therefore to brainstorm about existing experimental capabilities and platforms and the future needs that this group will particularly require. The two half-day sessions were organized around a set of invited presentations that triggered discussions about needs, advances and culminated in discussions on a set of early experiments which would leverage the upgrade capabilities both in the short term and enhance them in the longer term. The several steering talks were focused on introducing evolving techniques and needs.

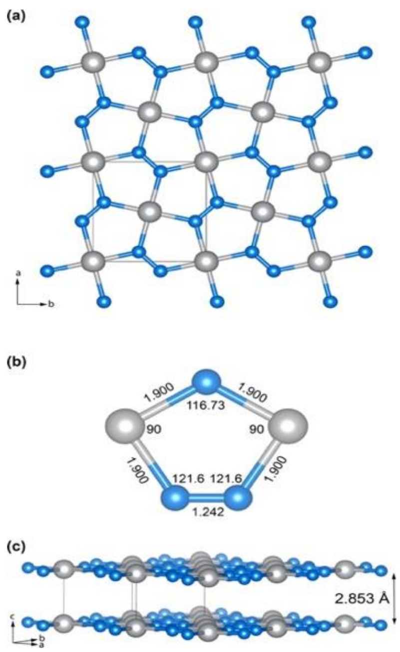


Figure 15: crystal structure of p-NiN_2 (a pentagonal 2D material with a tunable direct band gap) synthesized at high P-T conditions. Figure reproduced from (Bykov, Bykova et al. 2021)

Use of a rotational DAC (RotoDAC) for synthesizing novel alloys *(Arun Devraj, PNNL)* was reviewed by Arun Devraj. His talk explained in detail the complex experiments undertaken at HP-CAT in collaboration with Changyong Park in adapting and validating the RotoDAC developed in collaboration with Stas Sinogeikin of DACTools. Several research groups have been focusing on the use of RotoDAC for answering questions related to the role of shear in materials synthesis and shear-driven transformations (Levitas, Ma et al. 2012) (Huang, Shiell et al. 2024). Some of the outstanding questions remaining to be resolved are: experimentally characterizing the shear state of the sample, and understanding the role of shearing rate, temperature, dislocation pileup and chemical inhomogeneity in the formation of new materials. The current in situ experimental capabilities cannot address these questions, as most characterizations remain ex situ*,* performed after the retrieval of quenched sample. It was recognized that multiple probes need to be applied at sub-micron length scales to explore these parameters. For example, with the upgrade, XRD-XAS with a sub-micron, focused beam is expected to be available at 16ID-E and therefore, simultaneous diffraction and XANES mapping at mSec timescales could be used. Similarly, fast spatial mapping with a sub-micron focused, white-beam Laue diffraction in 16BM-D could be used to understand the stress-state of individual grains across the sample.

Crystal structure determination methods for advanced materials *(Stella Chariton, U Chicago)* In an insightful presentation, Stella Chariton reviewed the utility of combining multi-crystal diffraction measurements and DFT calculations that helped solve complex problems which would have otherwise been impossible (Bykov, Chariton et al. 2019) (Bykov, Bykova et al. 2021). In both the cases, the data quality is hampered by the fact that the samples were multi-crystalline and highly textured, as is normally the case with synthesis using LHDACs. While many groups have used theoretical simulations as a tool to identifying the phases (Walsh and Freedman 2018), the examples presented by Stella used more recent multi-crystal data analysis techniques offered by Crysalis^{Pro}, ShelXT (Sheldrick 2015) and Jana2006 (Petříček, Dušek et al. 2016). The data so obtained could be used in conjunction with first principles DFT simulations to arrive at totally unexpected results which could not have been otherwise possible. With the availability of higher brilliance and smaller focal spot size, coupled with more efficient detectors, the need for better data evaluation and analysis strategies was highlighted. The discussions that followed this insightful presentation agreed that use of on-board edge computing will help in more efficient data collection strategies (Manna, Loeffler et al. 2022) (Kandel, Zhou et al. 2023), including the collection of sufficient redundant data to validate the structural models, especially when mapping the post-laser-heated sample assembly. Strategies such as those discussed in the presentation need to be disseminated to the wider user community to advance the field to the next level.

In-situ transport measurements in a Paris-Edinburgh Cell *(Innocent Ezenwa, ANL)* was a presentation geared towards introducing the recent developments in the Paris-Edinburgh cell located at the 16BM-B station. The existing facility for measuring ultrasonic sound velocities, viscosities by radiographic imaging, densities and PDFs of amorphous materials is being augmented during the current upgrade with valuable additions such as a 1.2 M long horizontal focusing mirror, a vertically deflecting multilayer condenser, and an advanced white beam energy dispersive diffraction setup with a sophisticated collimation capability. In addition, as Innocent presented, the capability to measure resistivity and thermopower have been added. In collaboration with Curtis Kenny-Benson and Tyler Eastmond, extensive FEA simulations and field trials have managed to optimize sample assemblies and reproducibility. Moreover, an improved geometry considering electric field effects has allowed them to make reliable resistance measurements and seamlessly connect with ambient measurements of metals such as Bi, Pb, Sn. Efforts are underway to enlarge the scope of the resistance measurements with the addition of a complex impedance bridge to include non-metals. Measurement of thermopower and dielectric measurements at elevated pressure-temperature conditions has been a long-standing dream of the materials science community (Baker, Kumar et al. 2017) (Ahart, Somayazulu et al. 2008) (Wu and Cohen 2005) (Baker, Park et al. 2021) and this presentation enthused the workshop attendees about the possibility of combining many techniques in the PEC. Efforts are being made to extend the pressure range by employing polycrystalline sintered diamond as anvils, and to have better controls of temperature using tailored heaters.

Extending pressure, temperature, shear, and strain rate extremes and combining synthesis and validation of high-entropy materials in a large volume cell was discussed in a presentation by Prof. Yogesh Vohra of UAB. He emphasized the need for validating the superior elastic properties of several additively manufactured high-entropy materials that can only be performed effectively in a PEC (Hrubiak and Sturtevant 2023) (Pope, Iwan et al. 2023). Prof. Vohra emphasized the need for combining DAC and PEC experiments to understand the interplay of length scales and macroscopic, elastic properties of 2D and 3D additively manufactured materials.

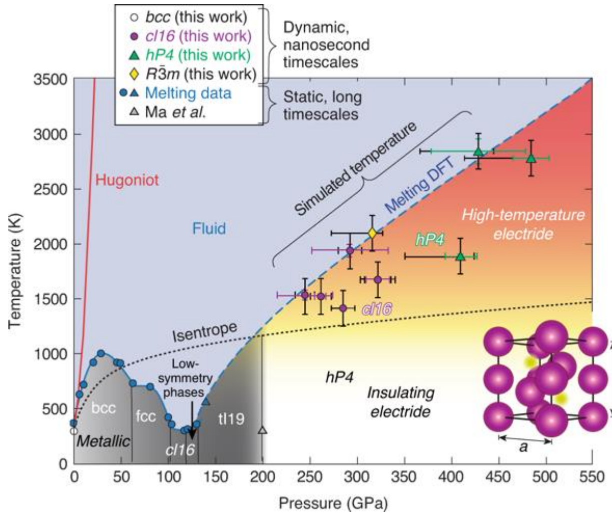


Figure 16: The high-pressure phase diagram of Na based on laser-driven ramp compression data showing the complex phases that occur under extreme pressure-temperature conditions. Figure reproduced from (Polsin, Lazicki et al. 2022).

In a similar strain, Prof. Russell Hemley of UIC highlighted the outstanding problems in extreme conditions science emphasizing the need to use a confluence of probes to understand, for example, *'kilovolt chemistry'* at TPa pressures where there exists no experimental data regarding the charge density in cold dense matter. He stressed the need for using coherence available after APS-U to enlarge the scope of imaging to understand high-pressure strength, plasticity, and rheology in materials. This experimental data from computed tomography and coherent diffraction imaging hitherto not applied to diamond anvil cells (Harder and Robinson 2013) (Husband, Hagemann et al. 2022) would become valuable in understanding how to predict properties of materials and augment existing databases of materials. These two presentations resulted in a broad range of discussions that encompassed the invited presentations of Amy Clarke (Col. Sch. Mines, *Advanced Characterization of Metals under Extreme Environments*) and Jason Jeffries (LLNL, *Overview of Materials Advances and Requirements for NNSA mission needs*). There was a general agreement amongst the participants that leveraging coherence, sub-micron beams, and AI and ML based data collection strategies is of paramount importance if the extreme conditions science community will be expected to address some of the outstanding problems awaiting to be solved and the advantages of APS-U can be best assimilated with the versatile diamond anvil cell. In fact, the presentation by Sakura Pasquarelli, Scientific Director of XFEL, added the importance of time-resolved XAFS and XANES measurements as a probe of chemical reactivity at elevated P-T conditions that also offers an exciting probe for melting studies (Pascarelli, McMahon et al. 2023) which is possible at HP-CAT given the upgrade plans of the spectroscopy beamline 16ID-D.

Summary of challenges and opportunities

The workshop recognized the need for enlarging experimental capabilities that particularly address defects and microstructures produced by pressure, shear, deformation, and strain rate and how they affect material response at extreme conditions and that, whether it be materials produced by novel processes like additive manufacturing or otherwise, the diamond anvil cell remains one of the best sample compartments. The enhanced coherence, focus and brightness assured by APS-U positions HP-CAT optimally to be able to achieve this. The technical advancements of diamond cells that include dynamic DAC (dDAC), rotational DAC (rotoDAC), and multi-axis DAC (MDAC), allow us to tailor the deformation mechanism and probe varying length scales using a combination of sub-micron probe beam and AI enabled mapping

techniques. Such measurements will not only bridge the micro to the macro scale but also augment meso-scale computational methods with valuable feedback.
The possibility of using broadband multilayer monochromators and MHz imaging detectors in dispersive spectroscopy opens the possibility of understanding chemical reactivity and electronic structure evolution at TPa pressures and 5000 K, a hitherto uncharted territory in condensed matter physics and reaction chemistry. The workshop felt that these studies will make major contributions to our understanding of corrosion, cavitation and chemical activity at extremes that is invaluable to our nation's energy plans that include nuclear energy.

The advances made in the PEC, in being able to perform multiple measurements on large mm sized samples at pressures up to 20 GPa (pressures of the order of 80 GPa have been recently reported with nanoPCD anvils (Le Godec, Dove et al. 2003), the technical maturity of the rotoPEC (Philippe, Le Godec et al. 2016), and the refinement of transport measurements in a PEC are all advances awaiting utilization for validating novel ferroelectrics and thermoelectric materials, understanding melt structure and compressed amorphous materials, and more importantly, scaling the discovery science originating from DACs to real-world applications.

Some of the challenging studies the workshop recommended, both as benchmarks and community engagement, included:

(1) PDF measurements and X-ray Raman measurements on glassy carbon to 2 Mbars.
(2) Exploring the enhancement of ferroelectric figure of merit in PbTiO3 class of ceramics at the pressure induced morphotropic transition(s).
(3) Combining in situ fast diffraction, x-ray imaging and thermal imaging to evaluate interplay between shear, temperature, and dislocations in a RotoDAC.
(4) Utilizing the double stage PEC to evaluate high entropy alloys made from 3D printing at Mbar pressures.
(5) Explore energy dependence of dissociation of H_2O, H_2O_2, NH_3, CO_2 and formation of intermittent chemical species.

Report on Breakout Sessions - 3

Understanding the EOS, transformations, deformations and bulk properties

Shanti Deemyad, University of Utah
Blake Sturtevant, Los Alamos National Laboratory
Rostislav Hrubiak, Dmitry Popov, HP-CAT-XSD, Argonne National Laboratory

This session was dedicated to exploring recent advancements in high-pressure studies aimed at unraveling the *complexities* of diverse materials under extreme conditions. Both experimental and theoretical topics were discussed, covering a wide range of high-pressure measurements conducted using various platforms such as the diamond anvil cell (DAC) and large volume press. These measurements were complemented by a diverse array of x-ray probes, including diffraction, spectroscopy, electron microscopy, and radiography, alongside other measurement techniques such as electrical, thermal, and ultrasound analysis. The overarching goal was to sample the latest experimental and theoretical efforts in understanding the fundamentals of material behavior, elucidating the correlation between structural information and bulk properties under extreme conditions, and to identify the technical challenges and opportunities at HP-CAT post APS-U.

The session covered a broad spectrum of topics, including:

- Elastic constants and material strength
- Non-hydrostatic compression and deformation behavior
- Equations of state (EOS) of complex materials
- Mesoscale modeling to understand the relationship between microscopic structure and macroscopic properties
- Kinetics and mechanisms of phase transitions
- Materials transport properties under extreme conditions
- Materials metastability and pathway-dependent properties with improved spatial and temporal resolution

The breakout session, conducted in two periods, featured presentations from 10 speakers, encompassing both theoretical and experimental aspects. To initiate the discussion, the session began with a presentation by HP-CAT staff highlighting upcoming measurement capabilities, particularly focusing on Laue diffraction in a DAC for observing microscopic phenomena and the large volume press capabilities for combined bulk measurements.

Subsequent discussion topics were presented by several invited speakers, each allocated 15-20 minutes for their presentations followed by open discussions. Summaries of the talks are provided below, grouped into the broader discussion topics listed above to facilitate organization. Finally, key technical challenges and opportunities identified by the presenters and ensuing open discussions, particularly in the post-APS-U era, are summarized at the end of this section. The breakout session was steered by several presentations which are presented in the order they were delivered along with a summary of discussions that these presentations generated.

Overview of HP-CAT Capabilities (D. Popov, R. Hrubiak, HP-CAT): After a brief introduction of the overall HP-CAT facilities, the focus was primarily on the capabilities of the 16-BM-B station at HP-CAT. This station features a large volume Paris-Edinburgh (PE) press system coupled with energy dispersive x-ray diffraction (EDXD). Prior to APS-U, 16BM-B beamline also had a setup for DAC measurements coupled with Laue diffraction. After APS-U, the Laue diffraction technique in the 16-BM-D station will be combined with the monochromatic diffraction technique in a way that a switch between monochromatic and polychromatic beams will be done in a matter of minutes without the need to realign the sample. The key capability provided by the combined application of these two techniques is that changes of both microstructure and atomic structure can be observed simultaneously providing comprehensive information on the mechanisms of compression using DACs and other platforms. Notably, the PE press setup's significant advantage lies in its larger volume compared to a DAC (2mm diameter by 2mm height), enabling the measurement of bulk phenomena such as equation of state (EOS), thermal expansivity, elastic properties, fluid/melt viscosity, amorphous density, liquid (im)miscibility, and thermal/electric properties, as well as materials synthesis. The capability to observe and quantify these phenomena in situ, alongside structural data, facilitates the correlation between microscopic and macroscopic material properties. Furthermore, combining monochromatic and Laue diffraction can be employed to quantify atomic scale phenomena like plastic deformation, product variants, interface propagation, and crystal growth. The presentation underscored the considerable potential for combining structural and in situ bulk properties measurements in the investigation of various materials systems, including, for instance, high-entropy alloys. Overall, the presentation provided an insightful overview of current HP-CAT capabilities and served as a foundation for discussing the current state-of-the-art and potential opportunities for future development.

Determining elastic constants at high pressure and temperature at beamline 16 BM-B; current capabilities and future opportunities (Blake Sturtevant, Los Alamos National Laboratory): Dr. Sturtervant's presentation delved into the pivotal role of elastic moduli in constructing equation of state and strength models, with a primary emphasis on enhancing measurement accuracy. Notably, Blake articulated a need for various technique advancements, citing the example of 3D rendering of samples using computed x-ray tomography in the PE press. This objective is important for reducing uncertainties associated with sound velocity measurements and the consequential derived values of materials' elasticity and EOS. The presentation underscored the usefulness and the critical influence of accurate ultrasound speed measurements using the PE press setup in 16-BM-B.

Tensorial stress, plastic strain fields in α - ω Zr mixture and transformation kinetics in diamond anvil cell *(*Achyut Dhar, K. K. Pandey, Valery I. Levitas. Iowa State University): Graduate student Achyut Dhar emphasized that obtaining all components of stress and plastic strain is key to understanding plastic-strain induced phase transformation quantitatively. The current method of using just volumetric strain-based energy density to evaluate the pressure has a fundamental flaw since presence of significant non-isotropic stresses, also requires the estimation of deviatoric strains. The axial x-ray diffraction is incapable of evaluating the deviatoric strains, and therefore, to address this shortcoming, a combined experimental-analytical-computational approach is proposed. This new robust framework aims to find all components of stress and plastic-strain tensors in the entire sample before, during, and after the phase transformation is complete. This new framework can be used for any material system, provided the material yields under stresses.

Importance of EOS in understanding pressure-induced phenomena (Wenli Bi, University of Alabama): Dr. Bi highlighted the crucial role of lattice information in comprehending pressure-induced emergent phenomena, including magnetism, superconductivity, quantum phase transitions, and quantum spin liquid. Dr. Bi advocated for the integration of x-ray diffraction with other probes such as x-ray spectroscopy, electrical transport, magnetization, and Raman spectroscopy to obtain comprehensive insights. Case studies, such as the tunable Weyl semimetal candidate $EuCd_2As_2$ and Se_2-containing compounds explored as quantum spin liquid candidates, underscored the importance of multimodal measurements. Dr. Bi emphasized the necessity for specialized sample environments for XRD, including lower temperatures, magnetic fields, and faster sample switching. The key takeaway emphasized the significance of combining various measurement techniques and the need for tailored sample conditions to advance our understanding of these complex materials.

Equation of state of polymers and related materials. (Muhtar Ahaiti, University of Illinois Chicago): Dr. Ahaiti's presentation centered on the overarching goal of determining the equation of state (EOS) for polymers and amorphous materials. He delved into the challenges associated with measuring the EOS, emphasizing the need for alternative methods and expressing a specific interest in low-pressure measurements to gain insights into polymer behavior. The presentation highlighted the significance of exploring diverse approaches to address experimental challenges in accurate PDF determination and EOS measurement, ultimately improving our understanding of the equation of state and the elastic properties of these materials. Dr. Ahaiti also emphasized the importance of future work, advocating for the integration of multiple probes such as sound velocity, Brillouin spectroscopy, and amorphous structural measurements with x-ray diffraction. Additionally, he underscored the value of attaining higher pressures for these probes, emphasizing a comprehensive approach to further advance research in this field.

Characterizing Phase Transformation and Twinning Variants in BCC Metals using Atomic Scale Simulations. (Phillip Tsurkan and Avinash M. Dongare, Materials Science and Engineering, and Institute of Materials Science, University of Connecticut): Graduate student Phillip Tsurkan presented his work on theoretical simulations of phase transitions in BCC metals. The different phase variants that form during Fe's pressure-induced α (BCC) ↔ ε (HCP) phase transformation influence the orientations present in the unloaded microstructure, which contribute to plasticity differently. These variants have been investigated using diamond anvil cell (DAC) experiments, where in situ x-ray diffraction provides a means to characterize the transformation behavior. However, due to the large area from which the diffraction signal is obtained, most studies have been limited to single crystal systems. This leaves the role of loading orientation and orientation of neighboring grains on variant selection unknown. For this reason, molecular dynamics (MD) simulations are employed to investigate microstructure evolution in polycrystalline Fe systems undergoing the α ↔ ε phase transformation. MD simulations can reproduce the phase transformation and defect evolution of Fe at an atomic scale. DAC conditions can be modeled, allowing for hydrostatic (and non-hydrostatic) loading and unloading, where "Virtual Texture Analysis" (VirTex) is used to characterize phase and twin variants. The advanced submicron Laue technique after the HP-CAT upgrade will allow for carrying out DAC experiments on polycrystalline Fe systems and characterize the variants present in individual grains to validate/improve the model. We can then use the improved model to investigate other effects on variant selections in Fe.

Negative stacking fault energy in FCC materials: Its implications. (D. You, O.K. Celebi, A.S.K. Mohammed, H. Sehitoglu, University of Illinois Urbana Champaign Mechanical Science and Engineering): The work presented by graduate student Daegun You is about an analytical framework capable of predicting the finite stacking fault width and critical stress for an arbitrary regime of stacking fault energy in FCC materials. It revealed dependencies of lattice constant, elastic constants, and unstable/intrinsic stacking fault energies on determination of dissociation stability, local and global equilibrium stacking fault, and CRSS. Therefore, precise determinations of lattice constant, elastic constant, and structure refinement under controlled conditions (such as deformation, shear) will help understanding the correlation between atomic structure, microstructure, and physical properties.

Anomalous Grain Growth in Zr During Phase Transformation at Room Temperature. *(*Raghunandan Pratoori, Iowa State University): Graduate student Raghunandan Pratoori's presentation was about grain growth in nanocrystalline materials during room temperature high pressure phase transformation, which is observed in various materials like Zr, InSb, Ba, Sr, Bi, Cu, etc. This is a phenomenon occurring at room temperature and the possibility of utilizing this method to obtain single crystalline phases makes it interesting to study this in detail. One hypothesis is that the grain growth occurs through a set of intermediate steps of low activation energy rather than the highly discussed recrystallization theory, which is supported by thermodynamics and kinetics calculations. These models can be tested/improved by time-resolved structural data during the grain growth in nanocrystalline materials.

Viscosity of hydrous magmas/melts in the Earth's upper mantle. (Jin Zhang, Texas A&M University) Dr. Jin Zhang discussed the Paris-Edinburgh (PE) press's unique capability for in situ high-pressure and high-temperature (P-T) viscosity measurements for broad user communities at HP-CAT. Drawing on her experience with in situ falling sphere viscometry experiments, Dr. Zhang outlined that there is substantial potential for future advancements to enhance this methodology. A primary focus involves exploring new sample capsule materials to better understand volatile storage and transport in deep planetary interiors. Current capsules, utilizing graphite and/or BN, face challenges related to inevitable volatile loss during experiments. Another avenue for improvement includes the implementation of smaller anvils in the Paris-Edinburgh press, facilitating higher-pressure experiments. This enhancement proves particularly advantageous for studying melt migration processes in the deeper regions of the Earth and other planets. Additionally, investigating technical developments for direct temperature measurements would be highly beneficial, addressing uncertainties associated with the current power curve method and potentially improving the accuracy of results. These considerations highlight the ongoing efforts to advance the capabilities of in situ high P-T viscosity measurements at HP-CAT.

Kinetic barriers in phase transitions and true phase diagram of materials. (Shanti Deemyad, University of Utah): Dr. Deemyad emphasized the significance of accounting for kinetic barriers in phase transitions to accurately determine the thermodynamic phase diagram of materials. The presentation delved into the specific case of lithium (Li) metal at elevated pressures and low temperatures. Different phases of Li can manifest under these conditions, depending on the specific pressure and temperature parameters chosen during the experiment. Dr. Deemyad underscored the importance of considering the optimal pressure-temperature (P-T) path rather than the easiest experimental path to attain the lowest energy thermodynamically stable phase. This approach helps mitigate discrepancies between experimental observations and theoretical predictions. Such considerations prove crucial for resolving contentious reports arising from high-pressure experiments and contribute to a more accurate understanding of material behavior. She further emphasized that exploring different thermal paths not only aids in determining the correct thermodynamic states of matter but also facilitates the synthesis of samples under appropriate experimental conditions. An illustrative example is the recent synthesis of single-crystal epsilon-iron achieved through an alternative thermal path.

Summary of challenges and opportunities

The session focused on diverse topics of high-pressure materials research, offering insights through a mix of theoretical and experimental studies. The outcome of the discussions underscores the imperative for collaboration across high-pressure disciplines, highlighting the needed combination of theoretical modeling and experimentation.

The presentations and the open discussions have identified some overlapping technical challenges and opportunities for improvements at the HP-CAT beamlines. The essential needs for the future experiments, i.e. the requested technical capabilities at HP-CAT, are summarized into several categories here:

Sample Environment Controls:

- Enhance capabilities for controls of various desired pressure-temperature (P-T) paths.
- Recognize the significance of lower temperature measurements, particularly for studying quantum materials.
- Design advanced sample environments for high-temperature measurements, encompassing melts, with considerations for reactivity and volatile containment.

Measurement Techniques:

- Implement 3D tomography for enhanced visualization.
- Assess the impact of strain measurements on solid-solid transitions. Advancements in rotational-DAC and Laue XRD.
- Quantify non-hydrostaticity, such as radial diffraction.
- Explore techniques for measuring or imaging crystal defects and dislocations, such as 3D XRD, coherent diffraction imaging, and ptychography.

Multimodal measurements Data Collection and Analysis:

- Establish collaborative platforms integrating measurements, molecular dynamics, and mesoscale modeling.
- Integrate automation for rapid sample alignment and scanning, incorporate AI-assisted parallel analysis, and implement feedback mechanisms.
- Emphasize the documentation of P-T paths and other experimental conditions in metafiles.

Addressing the challenges posed by multimodal measurements on samples, it was acknowledged that while difficult, such approaches hold promise for enhancing the precision of individual measurements. For instance, conducting multiple modes of measurement (e.g., across various HP-CAT beamlines) on the same loaded DAC under identical P-T conditions could improve experiment reproducibility by minimizing variations. The discussion also broached the notion of optimizing scheduling at HP-CAT to facilitate concurrent measurements across multiple beamlines, thereby streamlining experimental procedures.

Furthermore, the session featured presentations from both theoretical and experimental domains, each addressing overarching themes. This convergence prompted discussion on whether HP-CAT could assume a more central role in fostering collaboration between high-pressure theoreticians and experimentalists, thereby fostering collaborative platforms that integrate measurements, molecular dynamics, and mesoscale modeling. Such initiatives could catalyze interdisciplinary studies, driving forward our understanding of high-pressure phenomena.

Report on Breakout Sessions - 4

Advanced X-ray Capabilities

Andrew Krygier, Lawrence Livermore National Laboratory
Christopher Seagle, Sandia National Laboratory
Changyong Park and Yuming Xiao, HP-CAT-XSD, Argonne National Laboratory

With the enhanced brightness and coherence of the x-ray source at APS-U, the upgrade of HP-CAT beamlines (HP-CAT-U) leverages the improved APS-U source and will develop advanced high-pressure synchrotron capabilities to address a range of scientific questions under extreme conditions at various timescales from microseconds to hours. Associated with the improved x-ray source at APS-U, new developments in diagnostics and x-ray probing capabilities, many compatible with dynamic and static compression experiments, call for an increased level of collaboration and cooperation across the broader platforms of various beamlines and among the high-pressure users' community. The "Advanced X-ray Capabilities" session was aimed at identifying advanced complementary and supplementary x-ray techniques to address the highest priority scientific questions of matter under compression, static and dynamic, at HP-CAT-U and other APS-U beamlines. The covered topics include:

1. New advanced XRD-based techniques,
2. Advanced X-ray spectroscopy,
3. Bridging dynamic and static compression, and
4. Priorities and opportunities at APS-U and other large facilities.

New Advanced XRD-based Techniques (Changyong Park and Hau Zhou, X-ray Science Division, Argonne National Laboratory): The APS-U source will deliver high energy x-rays with approximately 500 times enhanced brightness, 20 times reduced horizontal source size, and 1000 times enhanced coherent fraction compared to the previous APS source. By fully matching the improved source, an experimental end station, 16-ID-E, at the HP-CAT will be newly commissioned to enable innovative high-pressure XRD experiments. The newly enabled XRD-based techniques include 2D scanning probe X-ray Diffraction Imaging (2D XDI) with sub-micron resolution, grain Bragg Coherent Diffraction Imaging (g-BCDI) for polycrystalline materials, and high frequency XRD for fast pressure- and temperature-ramp experiments. The XDI technique will be able to utilize 100 nm focused beam with pre-figured ellipsoid mirrors; the g-BCDI will combine a priori established multi-grain crystallography approach to enable a tracked probe of a targeted grain in polycrystalline sample while the sample's texture evolves with pressure; and the high frequency XRD will utilize the improved flux that is further boosted by a double multilayer monochromat or with 1-2 % bandwidth and a MHz frame-rate detector system. These XRD-based techniques are compatible with each other in terms of the instrumental requirements so that they can be integrated on the same experimental table. The orders of magnitude improved spatial and temporal resolution, compared to those from the previous APS source, will usher in a new frontier in studying pressure-induced solid-state processes and strain-rate dependent phenomena under various compression conditions.

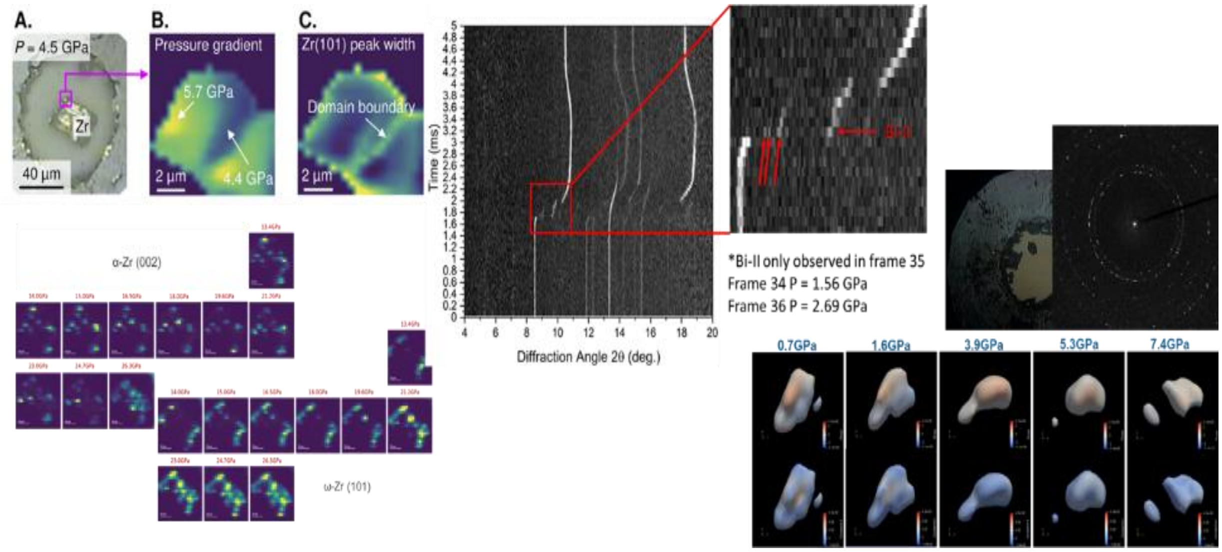


Figure 17: Figures illustrating 2D-XDI (left panel) (Hrubiak, Smith et al. 2019), high-frequency XRD (middle panel) and g-BCDI (right panel) techniques for high-pressure research. With APS-U beam's improved property, the spatial and temporal resolution can be greatly improved. Figures in the middle and right panel were adapted from (Husband, Hagemann et al. 2022), (Jenei, O'Bannon et al. 2018) and (Park 2024), respectively.

The Coherent High-Energy X-ray (CHEX) beamline at sector 28, one of eight feature beamlines of APS-U, will enable in situ, *real time* studies of materials synthesis and transformations using the enhanced coherence of the high energy x-ray beams (> 20 KeV). Coherent diffractive imaging (CDI) and X-ray photon correlation spectroscopy (XPCS) will provide transformative insight into materials structure, its heterogeneity and disorder, chemical and long-range interactions, dynamics, and evolution under real-world conditions and time frames. The CHEX beamline clusters multiplexed, simultaneously operating beamlines (400% user operation capacity at six experimental hutches) with complex apparatus for materials synthesis and transformation (e.g., high-pressure devices, molecular beam epitaxy (MBE), pulsed-laser deposition (PLD), metalorganic chemical vapor deposition (MOCVD), sputter, atomic-layer deposition (ALD), chemical vapor deposition (CVD), etc.). It takes the full advantages of high-energy x-rays that can be focused down to submicron scale, CdTe 2D detectors, rapid 3D volume acquisition of reciprocal space enabled by the coherent x-rays, and Fourier phase retrieval atomic mapping of local structures (e.g., coherent Bragg rod analysis or COBRA). This allows rendering electron density mapping with sub-angstrom resolution along the surface normal while resolving micron/submicron-scale features along lateral directions by scanning the probe over thin films and heterostructures. These capabilities will be useful to facilitate exploring emerging quantum behaviors under high-pressure conditions, for example, pressure-induced metal-insulator transition, superconductivity, etc.

Advanced X-ray Spectroscopy (Esen Alp, Yuming Xiao, and Daniel Haskel, X-ray Science Division, Argonne National Laboratory): There are several different inelastic X-ray scattering (IXS) methods, including Nuclear Resonance Spectroscopy (NRS), non-resonant IXS, resonant IXS (RIXS), X-ray Emission Spectroscopy (XES) and X-ray Raman Spectroscopy, which are suitable for studies under high pressure. Using these techniques, one can obtain rich information on materials such as phonon dispersion relations, elastic constants, speed of sound, anisotropy, phonon density of states, Debye temperature, recoil free fraction, vibrational entropy, specific heat, average atomic force constant and the resulting isotope fractionation, and melting point at extreme temperature and pressures (e.g., 4000 °C and 3 Mbar). Such properties of materials that can be measured at high pressures are relevant in solid state physics like in quantum phase transitions, in materials science, for example, discovering new materials for superconductivity, and in mineral physics, geophysics, geochemistry, and bioinorganic chemistry. They are useful to study structural phase transitions, valence transitions, spin transitions, element sensitive magnetism in rare-earth/Fe systems, mechanisms of magnetocaloric effect, and invar behaviors.

At the APS Sector 3, a short period superconducting undulator will be newly employed to compensate the reduction of electron beam energy from 7 to 6 GeV accompanying the APS

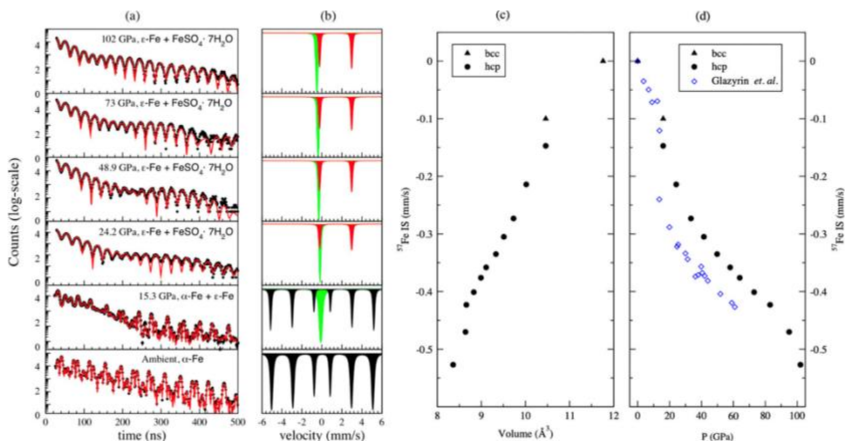


Figure 18: a) Nuclear forward scattering data of pure iron metal under pressure up to 102 GPa, b) Simulated data based on analysis of the data presented in (a), c) isomer shift of iron metal as a function of volume, and d) as a function of external pressure. Figure reproduced from (Desmarais, Bi et al. 2021).

upgrade. With reduced electron beam emittance, it will be possible to get sub-micron beam to probe matter at even higher pressures (e.g., P> 3 Mbar) more effectively and be less affected by pressure and temperature gradients. Micrometer focused beam will enable detailed analysis of heterogenous rocks such as meteorites and asteroid return samples.

At HP-CAT's high-pressure X-ray spectroscopy beamline (16-ID-D), XES, resonant and non-resonant IXS, and NRS techniques are available. Along with the APS upgrade, a new revolver

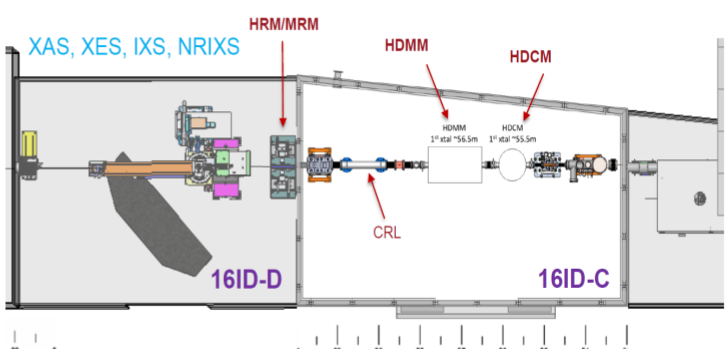


Figure 19: Schematic view of 16 ID-C/D hutches after HP-CAT-U. New HDCM, HDMM and CRL are located in ID-C; table-top high- and medium-energy resolution monochromators (HRM/MRM) will be located near sample stage in ID-D. Possible techniques include XAS, XES, IXS and NRIXS.

undulator with U21 and U25 will be employed together with a horizontally deflecting double-crystal monochromator (HDCM) and a double-multilayer monochromator (HDMM). The upgraded beamline will provide x-rays in the range of 4.8-60 keV with focus size down to sub-micron at sample. The photon flux on the sample will be increased by ~5-10 times compared to the past. When the multilayer optics (HDMM) is used, it will provide even higher flux by ~2 orders of magnitude, which makes fast non-resonant XES measurements (5-10 Hz) possible with a von Hamos spectrometer. Low temperature IXS under high pressure to study dynamics of superconducting materials, such as metal hydrides, will become possible with a specially designed cryostat.

Other than IXS based spectroscopy, the Polarization modulation spectroscopy (POLAR) beamline at the APS Sector 4, one of eight feature beamlines of APS-U, leverages the enhanced brightness of APS-U beams to probe, tune, and control electronic correlations in quantum and functional materials under simultaneous extreme conditions of pressure (multi-Mbar), high magnetic field (up to 9 Tesla), and low temperature (down to 1.4 K) using multi-modal probes (Raman spectroscopy and XRD) alongside x-ray dichroic measurements. The reduction in beam emittance by more than x100 results in as much flux gains for focused x-ray beam sizes in the 100-400 nm range. These beams allow probing electronic inhomogeneity in disordered or multi-phase systems by real-space mapping with dichroic contrast. Electronic correlations (magnetic, polar, chiral, orbital, nematic) lead to broken symmetries in electronic degrees of freedom, which can be sensed by measuring x-ray dichroism, i.e., polarization dependence in scattering and absorption cross sections at electronic resonances. Pressure and magnetic fields enable tuning of magnetic (exchange) interactions, electronic hybridization, electronic valence, and crystal fields triggering electronic order (magnetic, ferroelectric, superconducting, nematic, Kondo lattice), electronic disorder (mixed/fluctuating valence, spin liquids, spin glasses), or competing/segregated phases at the nano/meso-scale. The ability to probe most types of electronic/magnetic order, including imaging electronic inhomogeneity, at extreme pressure conditions is unique to bright synchrotron radiation sources. Exploring electronic phases that emerge from the combined effects of correlations and topology is needed to advance our fundamental understanding of quantum matter and seeding new concepts for the development of low-power microelectronics and encrypted information technologies.

POLAR also leverages the more than x100 increase in coherent flux for dichroic ptychography measurements (lens-less imaging in reciprocal space) to map electronic domains with enhanced spatial resolution relative to real-space mapping. The on-axis electron injection and small dynamical aperture of the APS-U storage ring lattice enables use of small diameter round

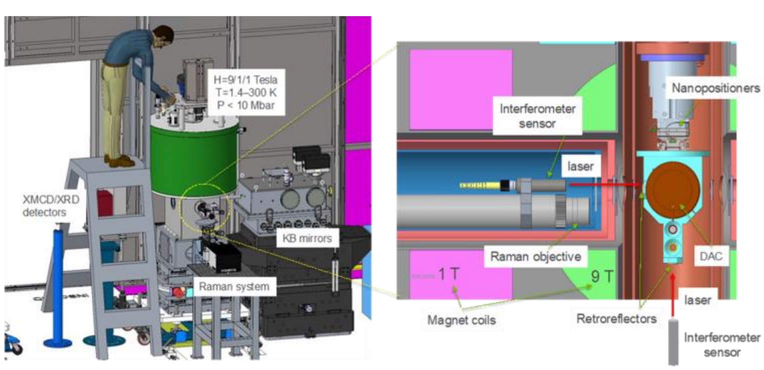


Figure 20: One of POLAR's primary instruments for HP dichroic measurements of electronic correlations in high magnetic field at low temperatures. The instrument allows multimodal Raman spectroscopy and XRD measurements. It uses interferometry and nano positioners to register (maintain) the illuminated region of the sample during real-space imaging (static) measurements, using 400 nm sized x-ray beams.

insertion device vacuum chambers and implementation of novel polarizing insertion devices to produce all polarization states (circular L/R, linear H/V, arbitrary linear, elliptical) directly at the source, in a much-expanded energy range (2.8-27 keV) and with large flux gains relative to x-ray phase plates, enabling heretofore impeded high-pressure studies of electronic correlations in 4d (Ru, Rh, …) and 5f (U, Np, Pu,…) elements using their high-energy K- and L- absorption edges, respectively.

Bridging Static and Dynamic Compression (Andrew Krygier, Lawrence Livermore National Laboratory): The laser-driven dynamic compression community uses many of the same or similar x-ray probe techniques that are widely used in static compression. Among these include x-ray diffraction and x-ray absorption spectroscopy, both of which are now being used at the National Ignition Facility (NIF), the Omega Laser Facility, and at the Dynamic Compression Sector (DCS) at APS (and APS-U), etc. There is much potential for collaboration between static and dynamic compression communities in both technique validation and comparison of results at different strain rates.

Strength of materials under various strain rate*:* There is much interest in determining the strength of materials in dynamic compression. One approach is to use x-ray diffraction in the (quasi-)radial geometry to directly measure the strain anisotropy intrinsic to strong materials when dynamically compressed. This capability has now been proven in dynamic compression experiments at DCS. The longer-term goal is to improve our understanding of strength behavior in dynamic compression as well as to constrain molecular dynamics potentials and strength models. In general, the strength of materials is strongly driven by strain rate, and it was proposed that a static compression facility like HP-CAT could help inform development of these models by performing moderate strain rate deviatoric strain experiments using dynamic diamond anvil cells in the radial diffraction geometry.

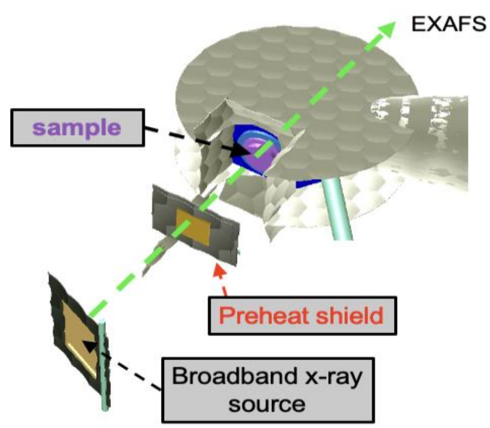


Figure 21: NIF EXAFS experiment configuration. X-rays are emitted by a laser heated foil (bottom left), penetrate the sample, and are collected by a high-resolution spectrometer (not shown). Data is used to determine temperature in ramp compression experiments at multi-Mbar pressure.

Measuring temperature from EXAFS*:* A major outstanding issue in dynamic compression experiments on solids is the nearly complete lack of bulk thermal diagnostics. This means that our understanding of the thermal state in materials experiments at Omega and the NIF is predominately informed by radiation hydrodynamics simulations and equation of state models. To address this, we have developed Extended X-ray Absorption Fine Structure (EXAFS) capabilities to measure samples ramp-compressed to multi-Mbar pressure. This data is then compared to predictions from finite temperature Quantum Molecular Dynamics (QMD) calculations to determine the temperature. By measuring EXAFS from solids at high pressures and 1000s K, a static compression experiment, e.g., at HP-CAT, could provide important validation of this approach as well as an experimental data-only temperature determination for NIF and Omega experiments.

Opportunities at Light Sources (Christopher Seagle and Tommy Ao, Sandia National Laboratories): A physics-based understanding of material response is required for predictive performance of materials and devices operating under extreme conditions of pressure and temperature. Construction and validation of physics-based models that provide this understanding typically require a combination of experimental approaches that probe different strain rate regimes (quasi-static to ~10^8/s) and different types of diagnostics that are sensitive to, for example, atomic, meso-, and bulk scale properties. A specific area of interest, that a static compression beamline like HP-CAT is well positioned to contribute, is in the *kinetics of phase transitions*. Polymorphic transitions, and solid-liquid/liquid-solid phase transitions are known to exhibit rate effects that depend on the detailed thermodynamics of the material system and the imposed strain rate that drives the system. To build predictive models of phase transition kinetics, experiments that probe material phase and phase fraction as a function of strain rate, ideally with time resolved diagnostics such as high frequency (up to MHz), x-ray diffraction are required. Several dynamic compression facilities across the NNSA complex are being utilized in this way, including HP-CAT using the dynamic DAC at ~10^3/s strain rates. Phase transitions are also being studied at high strain rates (up to 10^8/s) on dynamic compression facilities such as Z, NIF, Thor, and gas guns across the NNSA complex. However, key to interpretation of this dynamic data is a high accuracy understanding of the equilibrium phase diagram, as phase transition kinetic models are often grounded on the zero strain-rate (equilibrium) phase diagram and phase's internal energies. High accuracy equation of state and phase information as a function of pressure and temperature can be combined with dynamic phase information at varying strain rates to better understand phase transformation kinetics and validate/calibrate, and drive development of new response models. Material strength, damage, and ultimately failure is another topical area that would greatly benefit from a coordinated effort across a broad range of strain rates and conditions. For example, material strength, which can vary by orders of magnitude, dislocation generation, and dynamics all involve different mechanisms based on the crystal structure, strain rate, thermodynamic conditions, and often loading history. There is an opportunity to validate model predictions, and multi-scale strength physics models, through measurements of atomic to bulk scale response using a combination of static and dynamic drivers. HP-CAT has been a leader at applying radial diffraction at extreme conditions to measure deviatoric stress at extreme conditions; there is an opportunity to further refine this to higher strain rates and individual grains within the sample. New x-ray diagnostics, sensitive to strength, defects (dislocations), and material dynamics coupled to drivers capable of quasi-static to high strain rate loading will be required to fully resolve some of these questions.

With the APS upgrade, the higher flux and tighter focusing will greatly enhance opportunities in dynamic DAC and single-grain studies at extreme conditions. It also represents an opportunity for HP-CAT to further expand strain rate capabilities, both through dynamic DAC and pulsed power, the latter of which also represents an opportunity to combine atomic and bulk (continuum) response sensitive diagnostics in single experiment.

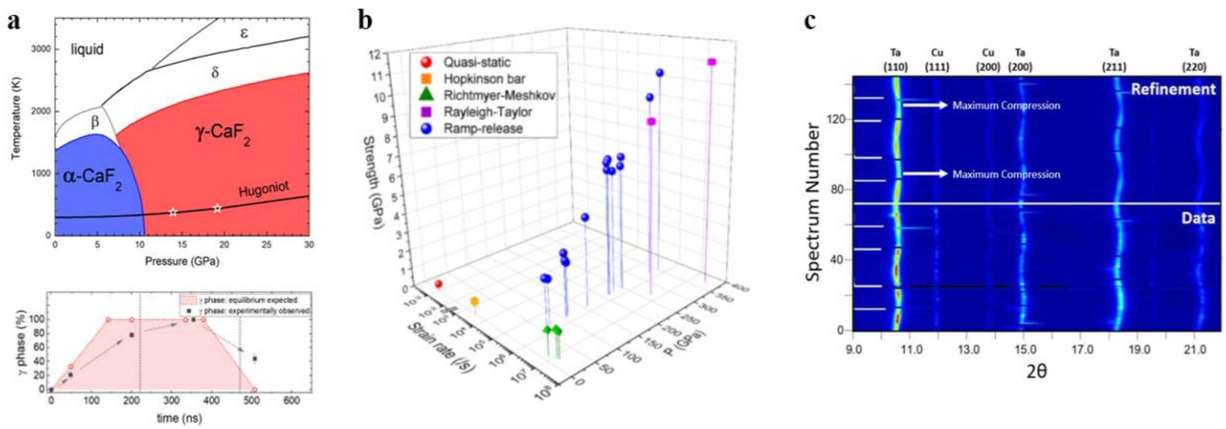


Figure 22: (a) Top: Calcium fluoride phase diagram with Hugoniot, Bottom: phase fraction observed in a dynamic compression experiment as a function of time compared to phase fraction expected from the equilibrium (no-kinetics) phase diagram (Kalita, Specht et al. 2023), demonstrating sluggish kinetics on nano second timescales. (b) Strength of tantalum over a large range of pressures and strain-rates demonstrating both significant strain rate and pressure effects on the strength (Prime, Arsenlis et al. 2022). (c) Radial diffraction of tantalum from HP-CAT which can be used to infer material strength at extreme conditions (Perreault, Huston et al. 2022). Figure in left panel reproduced from (Kalita, Specht et al. 2023), figure in center reproduced from (Prime, Arsenlis et al. 2022) and the figure in right panel, reproduced from (Perreault, Huston et al. 2022).

Pulsed power technology is well suited for studying the dynamic response of materials. Pulsed power drivers can efficiently compress large samples with unique uniformity and duration through complex loading paths to extreme pressure states. Accordingly, a midsize, pulsed power driver (e.g., Thor-200 machine) is an essential capability that could be explored as capability in the future at APS. One opportunity could be to implement a pulsed power capability first at an HP-CAT beamline by integrating Sandia's Veloce machine (e.g., at the 16-ID-E hutch). The Veloce machine is a compact pulsed power driver (3.6 m in width and 5.5 m in length) that delivers a 3 MA current pulse within 500 ns into a parallel plate load to magnetically compress samples up to 20 GPa. The following figure presents the coupling of the APS-U x-ray beam to a ramp compressed sample on the Veloce machine that would capture multiple XRD images during a single experiment. This would enable the investigation of key materials science questions, such as the role of microstructure, phase transition kinetics, and rate dependence of melting. To accommodate a Veloce machine in limited hutch space, reconfiguration of Veloce could be developed to reduce its size and enable easier operation and maintenance. Integration of such a capability at HP-CAT would extend the pressure-density regime beyond DAC regime to understand mesoscale material dynamics.

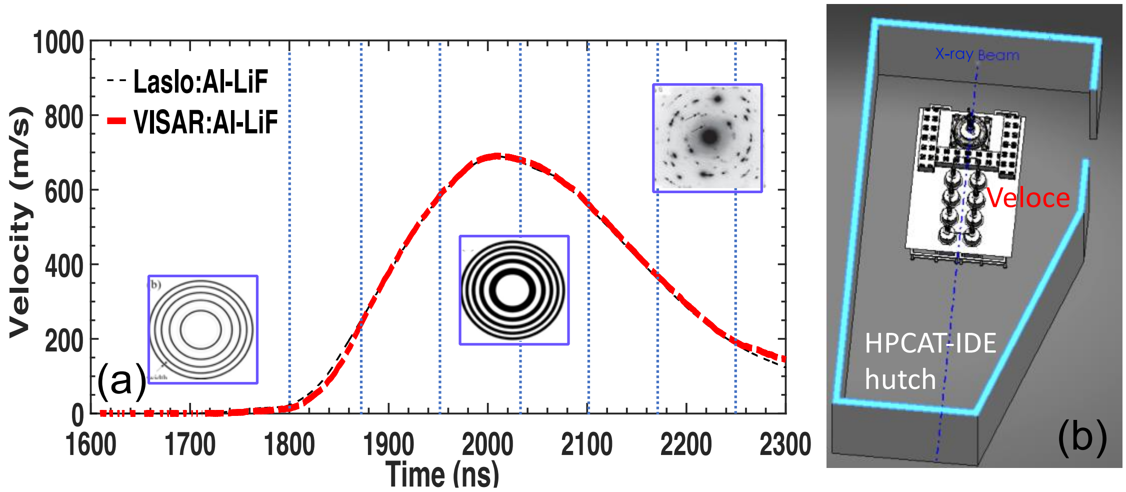


Figure 23: (a) Coupling of the APS-U x-ray beam to a ramp compression XRD experiment on the Veloce machine. (b) Schematic layout of the Veloce type machine and feasibility in incorporating within the 16-ID-E station.

Summary of challenges and opportunities

While advanced XRD capabilities are being developed at HP-CAT-U, there are a number of new and advanced x-ray capabilities emerging at other specialized beamlines at APS-U, such as Polarization Modulation Spectroscopy (4-ID POLAR), X-ray Photon Correlation Spectroscopy (8-ID XPCS), High-Energy X-ray Microscopy (20-ID HEXM), Coherent High-Energy X-ray (28-ID CHEX), and Atomic and 3D Micro and Nano Diffraction (34-ID ATOMIC and 3DMN) beamlines among a.k.a. “APS-U Feature Beamlines”. Many current capabilities, including Sector 13 GSECARS, 3-ID, 11-ID, 25-ID, 30-ID, and Sector 35 DCS-CAT beamlines will also be further enhanced with the improved x-ray source of APS-U; in particular, the faster x-ray probes will enable measurements of materials properties under various strain rates, and the finer resolution probes will obtain information from the individual grains within the sample. Conditions for high-pressure research are ripe at these beamlines and new opportunities are emerging for entire high-pressure community. The above recognized scientific and technical challenges will have better chances to be addressed with these enriched repertories of new experimental techniques. The development of HP-CAT-U will have to keep pace with these new developments and be closely coordinated with the other beamlines at the APS-U.

Report on Breakout Sessions - 5

Platforms for extending Pressure, Temperature, Shear, and Strain rate Extremes

Blake Sturtevant, Los Alamos National Laboratory
Yanbin Wang, SEES at Sector 13, University of Chicago
Tyler Eastmond and Guoyin Shen, HP-CAT, XSD, Argonne National Laboratory

The session focused on the development and implementation of techniques necessary to characterize materials in increasingly extreme sample environments. Techniques relevant to both diamond anvil cell (DAC) and large-volume press (LVP) platforms were discussed. Discussion topics included extended pressure-temperature (P-T) ranges, rate controls in heating/cooling and compression/decompression techniques, shear stress at high pressure, larger sample volumes, and optical access in LVP platforms. With the upgrades of both APS and HP-CAT underway, many of the discussions centered on leveraging the new capabilities to achieve future technical and scientific objectives. Each discussion was initiated with a short presentation from a subject matter expert who reported on the current state of research within their field, as well as several challenges associated with advancing the associated technical capabilities. This report aims to summarize the key features of each presentation as well as key points from the ensuing discussions.

Achieving Higher Pressures in a Diamond Anvil Cell (Zsolt Jenei, Lawrence Livermore National Laboratory): Conventional DAC experiments are limited to ~400 GPa, but new developments in toroidal DAC and double stage DAC are capable of pressures > 500 GPa (Dewaele, Loubeyre et al. 2018) (Dubrovinsky, Dubrovinskaia et al. 2012) (Jenei, O'Bannon et al. 2018). Dr. Jenei reported recent developments at LLNL using toroidal diamond anvils (Jenei, O'Bannon et al. 2018), shown in Fig. 24, fabricated by using a focused ion beam (FIB) to mill the culets of conventional single beveled diamond anvils. Case studies to test the design were conducted at HP-CAT and include calibrating the equation of state (EOS) of silver to > 4 Mbar (O'Bannon, Lipp et al. 2021), achieving better than 1% accuracy in bismuth EOS measurements (Campbell, Sneed et al. 2023), and accurately determining complex low symmetry crystal structures in dysprosium (Sneed, Söderlind et al. 2022).

One of the challenges in probing samples at > 4 Mbar pressures is the small sample size. The maximum pressure only exists in a central region of a few micrometers in diameter. Before the upgrade, the incident x-ray beam, typically 1-3 μm full width at half maximum (FWHM), is associated with a tail that could be wider than the central region of the highest pressure. This could result in unwanted diffraction from areas where severe pressure gradient exists. A similar challenge is related to laser heating samples at > 4 Mbar. The heating laser beam is often larger than the gasket hole, which results in heating the gasket material and weakening its strength, causing a pressure drop after laser heating. Other challenges include developing methods for full stress state characterization, providing sufficient x-ray flux for probing low-Z materials, and obtaining optimal anvil designs for reliably reaching pressures > 400 GPa and for ultrahigh pressures in the TPa range. The improved capabilities of HP-CAT after the upgrade will address these challenges. A submicron and brighter beam will be conducive to studying small samples as well as low-Z materials. An improved laser heating system will match the sample size to avoid unwanted heating of surrounding materials. Additionally, the improved x-ray techniques should allow for full stress state characterization via sub-micron x-ray diffraction mapping, sub-micron multigrain diffraction mapping, and coherent diffraction imaging.

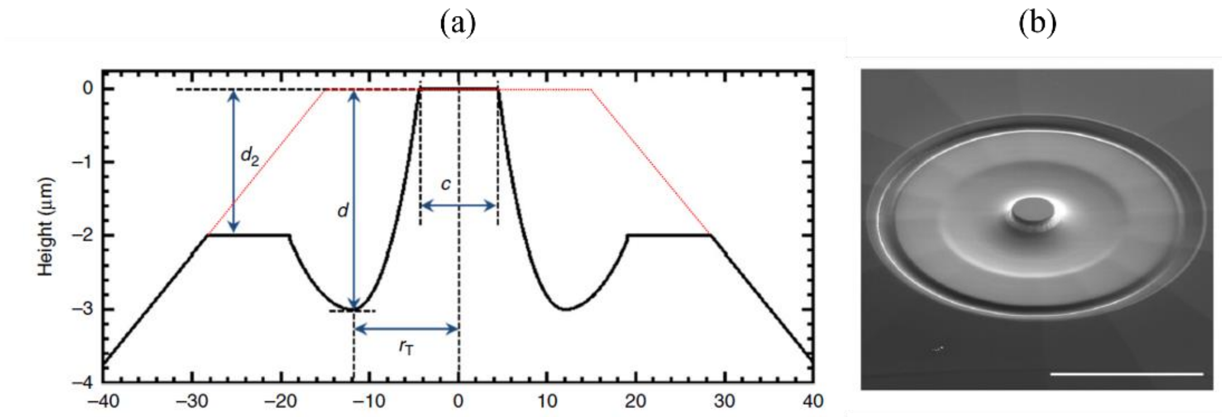


Figure 24: (a) Cross-sectional dimensions of a custom toroidal diamond anvil machined using FIB, with several variable geometric parameters. The red dotted line is the original profile of the cross section. (b) Image of the machined toroidal anvil, taken with a scanning electron microscope (scale bar is 30 μm). Figure adapted from Jenei et. al. (Jenei, O'Bannon et al. 2018).

Rate Control (Choong-Shik Yoo, Washington State University): Dynamic-DAC (dDAC) experiments allow for strain rates of ~10^3 GPa/s (Evans, Yoo et al. 2007) (Jenei, Liermann et al. 2019), bridging the gap between static and shock wave compression regimes and providing important complementary data. An important subject in dDAC studies is the effect of kinetics on the material response, in addition to the thermodynamics that dominate in the static experiments, illustrated in Figure 25. As an example, a recent study performed at the Extreme Conditions Beamline at PETRA-III used dDAC experiments to investigate the rate dependence of the III-V transition in bismuth (Husband, Hagemann et al. 2022). Various experiments were performed at rates between 0.01 to 780 GPa/s for powder and foil Bi samples with and without pressure transmitting medium. The results found a maximum over-pressurization for the Bi-III to Bi-V phase transition of 2.5 GPa as compared to static measurements, illustrating the importance of identifying and understanding kinetic effects in dynamic experiments.

An exciting opportunity for rate-controlled research would be the integration of MHz (up to 100 MHz) x-ray probes (XRD, X-ray imaging, SAXS) at the APS following the upgrade. The APS will operate in one of two modes, either 48- or 324-bunch, the latter with a bunch spacing of 11.4 nanoseconds. Coupled with increased brightness, up to 500 times, the storage ring itself would allow for data collection at a rate of ~87.7 MHz, if not limited by current detector technology. For example, the Adaptive Gain Integrating Pixel Detectors (AGIPD) at the European X-ray Free Electron Laser (EuXFEL) can resolve the x-ray pulses at a rate of 4.5 MHz (Allahgholi, Becker et al. 2019), which is an order of magnitude slower than the pulse rate at the APS. However, there are avalanche photodiode (APD) detectors that enable x-ray photon correlation spectroscopy (XPCS) at 100s of MHz. As detector technology continues to improve, it will make kinetic studies possible that are complementary to those at XFELs. For example, the undesired x-ray heating associated with XFEL experiments can be avoided at storage ring-based facilities such as APS-U. To enable the capability, the on-sample flux needs to be maximized by employing advanced x-ray optics, including those optics for pink x-ray beams. Faster MHz x-ray detectors will also be needed to make this possible. Implementing MHz x-ray probes could provide an opportunity to study kinetics under controlled ramping temperature and pressure conditions that may be used to mimic dynamic compression (e.g., an isentropic pathway). To do so, fast feedback would be needed on one of the thermodynamic parameters, requiring real-time pressure and/or temperature sensors with large signals so they could be measured in situ. Evolving material properties during the compression and heating process would also need to be factored in.

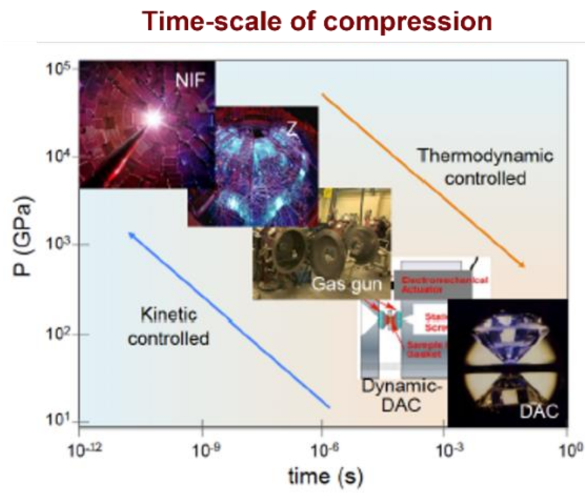


Figure 25: Various methods of high-pressure generation enabling static (thermodynamic controlled) to ultrafast dynamic events (kinetic controlled). Courtesy of Prof. Choong-Shik Yoo.

***Shear* Stress** (Arun Devraj, PNNL): Dr. Devaraj and his collaborators have developed a high-speed rotational DAC (HS-RDAC) that allows for the investigation of microstructural evolution of materials during shear deformation at high pressures (Devaraj, Liu et al. 2023). The setup was specifically designed for in situ characterization using synchrotron radiation. A principal focus of research using this technique has been solid phase processing of metallic alloys, e.g., lattice strain evolution and spatial variation of defect density have been observed in Cu-Ni materials (Park, Devraj et al. 2022).

Some of the existing associated challenges include microstructure gradients between the fixed and rotating diamonds in the HS-RDAC setup, as well as difficulties in quantifying the shear stress itself. Nano-scale 3D mapping on phase, texture, and strain distributions at various increments of shear strain could help with each of these, which will be possible after the APS upgrade.

Professor Vohra's group at UAB has focused extensively on studying high-entropy borides (HEB) at HP-CAT. One area of interest has been shear strength measurements of HEBs. A recent study on a (Hf, Mo, Nb, Ta, Ti) B_2 sample (Iwan, Lin et al. 2022) using radial diffraction in a panoramic DAC showed that the shear strength at 65 GPa approaches 8% of the shear modulus. To extend shear strength measurements beyond 100 GPa, x-ray transparent gaskets with higher strength and ductility need to be developed. Shear-induced structural distortions are also of interest (Sereika, Clay et al. 2023) and could be better understood with time-resolved RDAC diffraction studies.

Larger Sample Volumes (Malcolm Guthrie, ORNL): Due to the weak intensities inherent to neutron scattering techniques, the neutron scattering community has worked extensively to develop large-volume DACs, with sample volumes 10 times those used in typical x-ray DAC experiments at similar pressures. Dr. M. Guthrie and his collaborators at ORNL have recently had success in developing a megabar neutron DAC, with which they achieved a pressure of 1.15 Mbar in nickel using diamond anvils with 600 μm culets and a 400 μm diameter sample (Haberl, Guthrie et al. 2023). The ORNL team found it useful to provide both radial and axial support to their diamond anvils, as well as use CVD diamonds in place of natural diamonds.

At HP-CAT, a miniature multi-anvil device using diamond as anvils, called MDAC (Shen, Ferry et al. 2023) is being developed. The sample chambers for this setup have aspect ratios close to 1, a unique feature compared to existing DACs, with sample sizes varying from 100-400 μm^3. Pressures over 120 GPa have already been achieved using a prototype MDAC. The loading occurs in 3 orthogonal directions and can be controlled independently, which, when combined with the sample aspect ratio of 1, provides the possibility of fine control of differential stress and accurate measurements on strain. Thus, MDAC potentially enables deformation studies at pressures far exceeding what is currently achievable. The large volume DAC devices will be useful for additional post-upgrade studies as well, such as in studies of low x-ray scattering materials (hydrogen, amorphous materials, etc.), materials inhomogeneity, microstructure distribution, and transport property measurements that require thick voluminous samples. Technically, larger sample chamber allows for thicker insulating layers for laser heating, thus removing a key obstacle and providing effective and homogeneous heating in a laser heated DAC.

Existing challenges include the need for stronger seats and larger cells/drivers, due to the forces associated with increased sample volumes. This adds some complexity in terms of technical feasibility and cost. Implementing laser heating can also be challenging in neutron scattering experiments because a neutron beam is larger than the typical laser heating hotspot (Haberl, Quirinale et al. 2022), but this issue is largely mitigated for synchrotron x-ray experiments following APS-U due to the availability of a submicron beam.

Synthesis in a Paris-Edinburgh Cell (Yogesh Vohra, University of Alabama at Birmingham): Professor Yogesh Vohra and his team have not only characterized HEB using DAC studies at HP-CAT, but they have also synthesized them using the Paris-Edinburgh (PE) press. One example is the synthesis of $(Hf_{0.2}\ Ti_{0.2}\ Zr_{0.2}\ Mo_{0.2})B_2$ at 0.9 GPa and 1373 K, which was found to be stable up to 7.6 GPa and 1873 K (Iwan, Burrage et al. 2021). The importance of extending the P-T range of the PE press to P = 20 GPa and T = 3000 K was emphasized, as the stability field of synthesized materials could be more extensively probed in situ at these conditions. This could potentially be achieved with further development of a Cupped Drickamer Toroidal (CDT) cell, or using sintered diamond anvils, although the latter pose a challenge for heating at high temperatures.

Accessing Higher Pressures and Enabling Optical Access in Large Volume Presses (Yanbin Wang, GSECARS, The University of Chicago): High pressure experiments that allow for larger sample volumes are useful for material science that complements DAC experiments. Dr. Yanbin Wang presented some of the current efforts in the LVP community to extend the P-T range, as well as to allow optical access to the sample. A recent breakthrough (Ishii, Liu et al. 2019) was made in a Kawai-type MA setup in which WC anvils of grade TF05 and TJS01 (from Fujilloy) were implemented to reach pressures of 45 and 65 GPa, respectively, the latter being 2.5 times higher than the previous limit using conventional techniques. As shown in Figure 26, this was possible not only due to the increased hardness of the WC but because the anvils were tapered at 1° to reduce deformation of the truncated anvil face. Sintered diamond (SD) anvils (C2-grade) with 0.3° tapered edges and 1 mm truncated edge lengths have also been used to obtain pressures of 120 GPa in a Kawai-type MA setup. Efforts to increase the pressure range in PE press setups include implementing cupped-Drickamer-Toroidal (CDT) WC anvils (Kono, Park et al. 2014), SD anvils (Klotz, Hansen et al. 2019), and a double-stage cell with diamonds as the secondary anvils (Kono, Kenney-Benson et al. 2020).

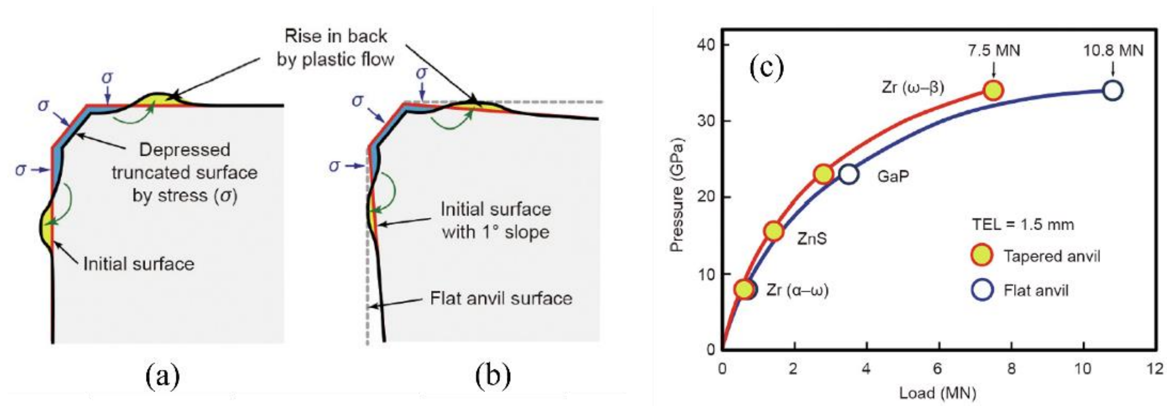


Figure 26: Effect of tapered anvil faces on pressure generation. (a, b) Comparison between the plastic deformation of a (a) non-tapered and (b) tapered anvil. (c) Improved pressure generation of tapered anvils over flat anvils. Figures reproduced from (Ishii, Liu et al. 2019).

There has also been a push to increase the temperature limits of LVP platforms. Recent advances in boron-doped diamond (BDD) heaters, in combination with SD anvils, allowed for P-T conditions of 53 GPa and 3300 K in a MA press (Xie, Chanyshev et al. 2021). BDD heaters also offer the advantage of being x-ray transparent, which can enable characterization techniques such as x-ray diffraction, radiography, and absorption. An existing challenge associated with LVP platforms is optical access to the sample. Benefits to this include measuring pressure in situ using ruby fluorescence, using Raman spectroscopy to characterize sample structure, or optically imaging material changes (Ryu, Kim et al. 2016). Possible avenues to achieve this include inserting optical fibers through sample cell gaskets, using diamond plugs in existing WC anvils, or implementing a transparent large anvil press (TLAP) (Sengupta, Ryu et al. 2012).

Integration of High-Pressure Devices to Large **Facilities (**Andrew Campbell, SEES and University of Chicago): While high-pressure research has been a vibrant application field at APS, there exist several opportunities and challenges in integrating of high-pressure devices at APS-U. Led by Prof. Andrew Campbell, the panel discussed a few aspects from a broad point of view:

- Facility upgrade goals do not always match experimental needs. We need to be mindful that the upgrade is designed for high brilliance, high coherence, and high energy. Other factors need to be considered for optical experimental designs, e.g., tight beam focus is not always optimal, synchrotron timing is not optimal for NRIXS, hard vs soft x-ray at other facilities.
- Increasing the efficient use of beamtime permits more experiments and greater number of users. However, it puts strain on beamline staff. Are there better modes for efficiency, including those of remote operation, mail-in service, or shared beamtime between user groups?
- Multimodal experiments can provide complementary analytical approaches simultaneously. What are the hurdles? Beamtime allocation calendar? Techniques/equipment? And/or support staff?
- Cross-facility management (e.g., SEES, NNSA-SSAA) offers potential to optimize experimental end station capabilities according to the source and scientific agenda.
- Software developments across communities are strongly emphasized in the panel.

Summary of challenges and opportunities

This breakout session discussed many of the future opportunities relevant to DAC and LVP platforms, as well as how the community can take full advantage of these opportunities through the capabilities provided because of the APS and HP-CAT upgrades. Some of the main opportunities identified during the session for extending pressure, temperature, shear, and strain rate extremes are as follows:

- A "clean" submicron beam, as well as an improved laser heating system, will reduce both unwanted diffraction and heating at the edges of the gasket hole during DAC experiments, enabling experiments for smaller samples and subsequently higher pressures.
- MHz x-ray probes will enable kinetic studies complementary to XFEL experiments, as the former avoids undesired heating of the sample. The upgraded storage ring would allow for ~88 MHz sampling rates, but improvements in detector technology are needed to fully realize this potential.
- A brighter x-ray beam will allow time-resolved rotational DAC studies to be used to study shear-induced structural distortions in various materials. Nano-scale 3D mapping of phase, texture, and strain distributions as shear strain is incremented will provide detailed information of the materials under shear.
- The large volume DAC (~400 µm sample size) developed at neutron facilities and the development of a multiple axis diamond anvil cell (MDAC, with sample volumes

from 100-400 μm^3) will provide means to probe larger samples at Mbar pressures, enabling studies of low-Z materials, material inhomogeneity, microstructure distribution, and transport property measurements.

- Further development of CDT or sintered diamond anvils for the PE press would allow for more extensive characterization of the stability field of synthesized materials.
- In addition to increasing the maximum achievable pressure in the PE press, some types of sintered diamond anvils would provide optical access to the sample. The implications of this would include Raman spectroscopy, optical imaging, and pressure measurement using ruby fluorescence.

The APS upgrade will bring with it an abundance of opportunities for high-pressure science, requiring communication and coordination between beamline staff and users. While higher x-ray flux, faster detectors, and multimodal experiments enable large amounts of data collection and a greater number of users, better modes of efficiency should be considered to avoid overstraining beamline staff. Collaborations across facilities and universities will be key to optimize end stations, develop software for data acquisition and analysis, and ultimately, push new scientific discoveries in the high-pressure community.

Closing Remarks

The HP-CAT workshop was a great opportunity to bring together our community and celebrate together the 20-year facility anniversary. The anniversary celebration was delayed (Nov. 2023 marked closer to ~22 years of continuous operations at HP-CAT) due to the global pandemic, that has impacted all of us. This workshop marked the first full onsite large audience event that we were able to host post-pandemic, and being able to reconnect and hear about the numerous achievements that have been accomplished across the research community was impressive and inspiring. From few-GPa pressure, that could be achieved with DAC in the early 1960s, today we are routinely able to achieve pressures of 300 GPa – even approaching TPa pressures with new double-stage and toroidal anvils concepts. Concurrently, laser heating and cryocooling capabilities have been developed by the community to couple DAC for high-P and mK to 4000+ K temperature measurements. Further advances in LVP static pressure devices, laser/gun shock platforms, and evolution of DAC (e.g. d-DAC, roto-DAC, etc.), have and continue to push the boundaries of extreme P, T, strain-rate and complex loading. From onset, static DAC and LVP platforms, in particular, have been closely aligned with evolution in large scale x-ray light sources. The intricate connection between developments in pressure generating devices and x-ray diagnostics have been mutually beneficial, and this connection is expected to be even closer going forward.

At the time of the workshop in November of 2023, APS was in the midst of a yearlong upgrade shutdown, which commenced in April of 2023. With the release of the workshop report, the APS-Upgrade to MBA lattice has been completed and first light is being introduced to experimental stations. On July 03, 2024, HP-CAT received its first x-ray beam post-upgrade in the 16-ID-A station and is on schedule to complete a comprehensive upgrade across all nine stations (five experimental and four optics stations) and various off-line laboratory equipment over the next two years. Higher brightness, coherence at higher energies, and lower emittance has been the *mantra* of the MBA lattice that replaced the APS ring which saw first light in 1990. While many beamlines may benefit from one or two of the above enhancements, HP-CAT and extreme conditions science in general will certainly have the opportunity to leverage all improvements from the new lattice. Coupled with quantum jumps in detector technology, focusing optics, AI/ML based data acquisition and pre-processing, one can now realistically think of routine studies at TPa pressures and 1000s K at the upgraded HP-CAT. While this extends static pressure measurements into P-T space hitherto unavailable, there is a need to enhance our toolkit that can leverage coherence and brightness more effectively. DCS, XFEL sources have opened newer vistas in time-resolved measurements. Similarly, laser facilities like Omega and NIF, as well as the pulsed power facilities like Z machine, offer much higher strain rates but understanding the microstructure and its consequent effect on material behavior is awaiting to be explored and is a valuable input in design of future materials. Precise measurements of P-V-T-s behavior in a range of materials can now be made given the HP-CAT-U capability to bridge length scales from the coherent imaging capabilities. Utilizing AI/ML capabilities to pre-process data quickly allows better experimental workflow management with sub-micron beams that interrogate grain boundaries and mixed phases. These studies will help unlock the potential of advanced manufacturing techniques in designing next-generation materials and validate their application potential. Similarly, accessing TPa pressures and temperatures more than 5000 K simultaneously expands the P-T space hitherto a purview of dynamic measurements. Dynamic DAC experiments and ramp heating experiments make it possible to access loading rates that are currently inaccessible.

The workshop served as the vehicle that not only engineered discussions on what can be achieved with the APS and HP-CAT upgrades, but also allowed the beamline scientists of HP-CAT and the user community to plan first experiments that commission these capabilities, benchmark them against existing capabilities and most importantly, adapt and evolve quickly so that the coming years will see the community at large progress rapidly.

Acknowledgements

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Workshop Agenda

Opportunities for Advancement of Studies of Matter at Extreme Conditions with APS/HPCAT-Upgrade

November 7-9, 2023

	Day 1 - Tuesday, November 7, 2023	
9:00 am – 9:45 am	Arrival and Registration / Badging	APS Atrium, Auditorium 402
9:45 am	Opening welcome by Nenad Velisavljevic, HPCAT Director	Auditorium 402
10:00 am - 11:30 am	**HPCAT 20year anniversary celebration** • Dr. Paul K. Kearns, ANL DirectorDr. Kimberly S. Budil, LLNL Director • Dr. D. Allen Dalton, NNSA-HQ Dir. Federal Program • Manager, Office of Experimental Sciences (NA-113) • Dr. Nenad Velisavljevic, HPCAT Director – HPCAT • history and look ahead • Dr. Laurent Chapon, APS Director – Update and • overview of APS and APS-Upgrade	Auditorium 402
11:30 am	**Anniversary Celebration – Cake cutting** Photos Buffet Lunch	Lower Gallery
1:00 pm	**Start of Scientific Program**	
1:00 pm	Dr. Nenad Velisavljevic, HPCAT Director - High-pressure research and workshop charge and goals	Auditorium 402
1:30 pm	Dr. Gregory B. Stephenson, Argonne Distinguished Fellow and Former APS Director – Challenges and opportunities with next generation APS-U beam	Auditorium 402
2:00 pm	Dr. Dana Dattelbaum, LANL program manager – Overview of NNSA light source work current and future opportunities, such as DMSS	Auditorium 402
2:30 pm – 2:45 pm	Break	
2:45 pm	Prof. Amy Clarke, Colorado School of Mines and Director of ACME2 SSAA Center - Overview of materials advances (new classes of advanced materials summary), challenges and needs for new x-ray diagnostics + new Advanced Characterization of Metals under Extreme Environments (ACME2)	Auditorium 402
3:15 pm	Dr. Jason Jeffries, LLNL Program Manager - Overview of materials advances and requirements (NNSA mission relevant) and needs for new x-ray diagnostics	Auditorium 402

<table>
<tr><td>3:45 pm</td><td colspan="2">Dr. Nathan Barton, LLNL Program manager – Strength models and high P-T data needs toward development of next generation predictive models</td><td>Auditorium 402</td></tr>
<tr><td>4:15 pm</td><td colspan="2">Break</td><td></td></tr>
<tr><td>4:30-
6:30pm</td><td colspan="2">Poster session</td><td>Lower Gallery</td></tr>
<tr><td></td><td colspan="2">Day 2 - Wednesday, November 8, 2023</td><td></td></tr>
<tr><td>8:00 am</td><td colspan="2">Prof. Andrew Campbell, U. Of Chicago and Director of SEES – Overview of new Synchrotron Earth and Environmental Science (SEES) and future plans at light sources, including sector 13 at APS</td><td>Auditorium 402</td></tr>
<tr><td>8:30 am</td><td colspan="2">Dr. Michael Walter, Director of Earth and Planets Laboratory, Carnegie Institution for Science – Future opportunities and needs with high pressure and synchrotron studies in support of Earth and Planetary studies</td><td>Auditorium 402</td></tr>
<tr><td>9:00 am</td><td colspan="2">Dr. Maddury Somayazulu, ANL-XSD/HPCAT - HPCAT-Upgrade overview</td><td>Auditorium 402</td></tr>
<tr><td>9:30 am</td><td colspan="2">Coffee Break and head to breakout sessions</td><td></td></tr>
<tr><td colspan="4">Breakout Sessions</td></tr>
<tr><td>10:00 am
–
12:00 pm</td><td>Session 1 – E1100
Extending the practical limits of pressure, temperature, and strain rate</td><td>Session 2 – E1200
Advanced Materials</td><td>Session 3 – Lower Gallery
Understanding the EOS, transformations, deformations, and bulk properties</td></tr>
<tr><td>12:00 pm</td><td colspan="2">Lunch</td><td>Lower Gallery</td></tr>
<tr><td>1:30 pm –
3:30 pm</td><td>Session 4 – E1100
Advanced X-ray capabilities</td><td colspan="2">Session 5 – E1200
Platforms for extending P, T, shear, and strain extremes</td></tr>
<tr><td>3:30pm</td><td colspan="2">Break</td><td></td></tr>
<tr><td>4:00 pm-
5:30 pm</td><td colspan="2">Tour of HPCAT facilities AND Breakout session leads work on note capturing</td><td>HPCAT, Sector 16
Building 434E</td></tr>
<tr><td>6:00 pm</td><td colspan="2">Banquet Dinner</td><td>ANL Guest House</td></tr>
</table>

	Day 3 - Thursday, November 9, 2023	

<table>
<tr><td>8:00 am</td><td colspan="4">Dr. Sakura Pascarelli, Scientific Director at European XFEL – Overview of XFEL and opportunities with XFEL and Synchrotron light sources</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>8:30 am</td><td colspan="4">Dr. Peter Celliers (one of LLNL lead scientist on NIF ICF experiments) – Overview of recent NIF ICF success and further data needs from HPCAT and other platforms</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>9:00 am</td><td colspan="4">Dr. Chris Seagle, SNL Dynamic compression group manager - Pulsed power and dynamic compression experiments and opportunities for further understanding of materials at extreme P-T and strain rates (also discuss difference/connection between static and dynamic and the evolving pulsed power user program)</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>9:30 am</td><td colspan="4">Mathew Cherukara, ANL-XSD Computational X-ray Science Group Leader – Big data science and ML developments at APS-ANL and application for x-ray sciences</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>10:00 am</td><td colspan="4">Coffee Break and head to breakout sessions</td><td colspan="2"></td></tr>
<tr><td colspan="7">Breakout Sessions</td></tr>
<tr><td>10:30 am – 12:30 pm</td><td>Session 1 –
E1100

Extending the practical limits of pressure, temperature, and strain rate</td><td>Session 2 –
E1200

Advanced Materials</td><td>Session 3 –
Lower Gallery

Understanding the EOS, transformations, deformations, and bulk properties</td><td colspan="2">Session 4 –
401-A5000

Advanced X-ray capabilities</td><td>Session 5 –
401-B2100

Platforms for extending P, T, shear, and strain extremes</td></tr>
<tr><td>12:30 pm</td><td colspan="4">Lunch</td><td colspan="2">Lower Gallery</td></tr>
<tr><td>1:30 pm</td><td colspan="4">FINAL report out by Breakout sessions chair</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>2:30 pm</td><td colspan="4">Dr. Nenad Velisavljevic and Dr. Maddury Somayazulu – Closing remarks</td><td colspan="2">Auditorium 402</td></tr>
<tr><td>2:45 pm</td><td colspan="4">Adjourn and Additional Tours of HPCAT</td><td colspan="2"></td></tr>
</table>

List of Figures

Figure 12: A comparison of the level of detail and information available from high-resoultion imaging. The composite image and corresponding plot on the left represent a compression rate of ~54 TPa/s, collected at a rate of 4 kHz (PETRA-III) while the composite image and corresponding plot on the right represent a compression rate of ~61 TPa/s, collected at a rate of 560 kHz (Eu-XFEL). The detail, density, and even oversampling of data has the potential to reveal minute or short-lived phenomena during and/or immediately following sample compression.

Figure 13: Modeling the axial heat flow and temperature distribution through a sample volume as a function of laser heating pulse duration suggests there is an optimum time range – in this case, on the order of a few tens of nanoseconds – to reach maximum temperature throughout the sample volume while minimizing some of the deleterious effects of melting and reactions that can result from continuous heating at very high temperatures in the laser heated DAC (Goncharov, Prakapenka et al. 2010). Figure reproduced from (Goncharov, Prakapenka et al. 2010).

Figure 14: The RotoDAC, TomoDAC and MDAC are three variants of the cells designed at HP-CAT for leveraging APS-U and performing measurements that can highly benefit studies on discovering advanced materials and exploring the recovery pathways.

Figure 15: crystal structure of p-NiN_2 (a pentagonal 2D material with a tunable direct band gap) synthesized at high P-T conditions (Bykov, Bykova et al. 2021). Figure reproduced from (Bykov, Bykova et al. 2021).

Figure 16: The high-pressure phase diagram of Na based on laser-driven ramp compression data (Polsin, Lazicki et al. 2022) showing the complex phases that occur under extreme pressure-temperature conditions. Figure reproduced from (Polsin, Lazicki et al. 2022).

Figure 17: Figure 17: Figures illustrating 2D-XDI (left panel) (Hrubiak, Smith et al. 2019), high-frequency XRD (middle panel) and g-BCDI (right panel) techniques for high-pressure research. With APS-U beam's improved property, the spatial and temporal resolution can be greatly improved. Figures in the middle and right panel were adapted from (Husband, Hagemann et al. 2022), (Jenei, O'Bannon et al. 2018) and (Park 2024), respectively.

Figure 18: a) Nuclear forward scattering data of pure iron metal under pressure up to 102 GPa, b) Simulated data based on analysis of the data presented in (a), c) isomer shift of iron metal as a function of volume, and d) as a function of external pressure. Figures reproduced from (Desmarais, Bi et al. 2021).

Figure 19: Schematic view of 16 ID-C/D hutches after HP-CAT-U. New HDCM, HDMM and CRL are located in ID-C; table-top high- and medium-energy resolution monochromators (HRM/MRM) will be located near sample stage in ID-D. Possible techniques include XAS, XES, IXS and NRIXS.

Figure 20: One of POLAR's primary instruments for HP dichroic measurements of electronic correlations in high magnetic field at low temperatures. The instrument allows multimodal Raman spectroscopy and XRD measurements. It uses interferometry and nano positioners to register (maintain) the illuminated region of the sample during real-space imaging (static) measurements, using 400 nm sized x-ray beams.

Figure 21: NIF EXAFS experiment configuration. X-rays are emitted by a laser heated foil (bottom left), penetrate the sample, and are collected by a high-resolution spectrometer (not shown). Data is used to determine temperature in ramp compression experiments at multi-Mbar pressure.

Figure 22: 22(a) Top: Calcium fluoride phase diagram with Hugoniot, Bottom: phase fraction observed in a dynamic compression experiment as a function of time compared to phase fraction expected from the equilibrium (no-kinetics) phase diagram (Kalita, Specht et al. 2023), demonstrating sluggish kinetics on nano second timescales. 22(b) Strength of tantalum over a large range of pressures and strain-rates demonstrating both significant strain rate and pressure effects on the strength (Prime, Arsenlis et al. 2022). 22(c) Radial diffraction of tantalum from HP-CAT which can be used to infer material strength at extreme conditions (Perreault, Huston et al. 2022). Figure in left panel reproduced from (Kalita, Specht et al. 2023), figure in center reproduced from (Prime, Arsenlis et al. 2022) and figure in right panel reproduced from (Perreault, Huston et al. 2022) is acknowledged.

Figure 23: (a) Coupling of the APS-U x-ray beam to a ramp compression XRD experiment on the Veloce machine. (b) Schematic layout of the Veloce type machine and feasibility in incorporating within the 16-ID-E station.

Figure 24: (a) Cross-sectional dimensions of a custom toroidal diamond anvil machined using FIB, with several variable geometric parameters. The red dotted line is the original profile of the cross section. (b) Image of the machined toroidal anvil, taken with a scanning electron microscope (scale bar is 30 μm). Figure reproduced from Jenei et. al. (Jenei, O'Bannon et al. 2018).

Figure 25: Various methods of high-pressure generation enabling static (thermodynamic controlled) to ultrafast dynamic events (kinetic controlled). Courtesy of Prof. Choong-Shik Yoo.

Figure 26: Effect of tapered anvil faces on pressure generation. (a, b) Comparison between the plastic deformation of a (a) non-tapered and (b) tapered anvil. (c) Improved pressure generation of tapered anvils over flat anvils. Figures reproduced from (Ishii, Liu et al. 2019).

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