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High-Pressure Research at the Advanced Photon Source

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Introduction

Pressure, together with temperature, composition, or external fields, alone or in combination, can be used as principal variables in directing and controlling matter as well as for the synthesis of revolutionary new materials. The impact of extreme conditions to science, however, has been previously limited by the available vessels and probes. The last decade has witnessed a significant surge in extreme conditions science, thanks to the development of *in situ* synchrotron radiation (SR) probes and high-pressure (HP) instrumentation. Extreme conditions directly alter chemical, structural, mechanical, electronic, magnetic, and phonon properties of materials, revealing intriguing behaviors across boundaries between insulators and metals, ferromagnets and superconductors, ordered and disordered, and vigorously reactive and inert compounds. In Earth and planetary sciences, research from simulating experiments at high pressure and high temperature provides key information in understanding processes, dynamics, and formation of the deep interiors. For materials applications such as the search for new energy materials and superhard materials, extreme conditions remain a vast, unexplored field.

Because HP conditions are achieved at the expense of diminishing sample volumes, the high-brilliance, high-energy, low-emittance SR sources provide powerful micro-sampling probes for the minute samples and resolve weak sample signals from the background signals of massive surrounding vessel materials. Thus, HP science becomes an important application field for SR sources. Research under extreme conditions is being conducted at many beamlines at the Advanced Photon Source (APS), including two dedicated HP beamlines, HPCAT (sector 16) [1] and GSECARS (sector 13) [2-4], and at least ten other sectors (1, 3, 4, 9, 11, 15, 20, 30, 32, 34) having major HP science programs. Among the array of X-ray techniques, structure determination using X-ray diffraction (XRD) remains the dominant one, while significant progress has been made in integrating other X-ray techniques for HP research, such as X-ray spectroscopy (XRS, absorption, emission), inelastic X-ray scattering (IXS), nuclear resonant scattering (NRS), and nanoimaging techniques.

In this article, we present several recent examples of research conducted at APS beamlines in the areas of physics, chemistry, materials science, and Earth and planetary sciences, followed by a brief introduction

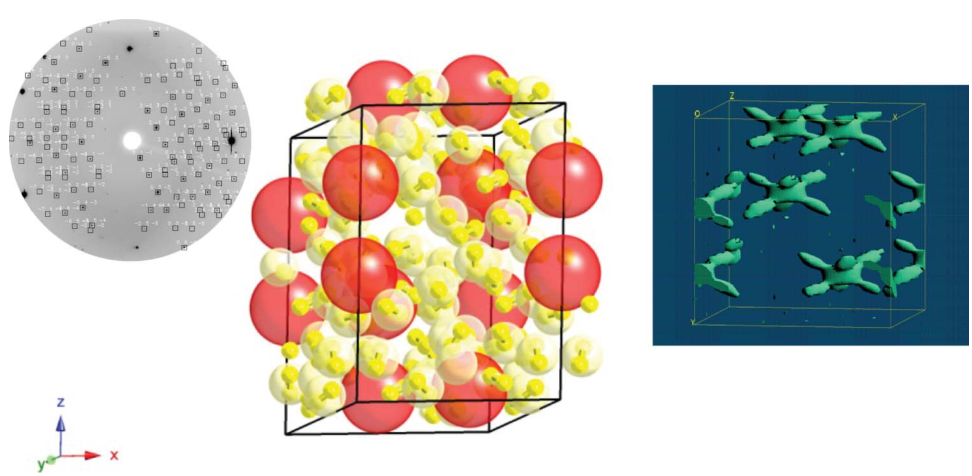

Figure 1: The compound $Xe(H_2)_7$, which forms at 5.9 GPa, remains stable at pressures in excess of 255 GPa.

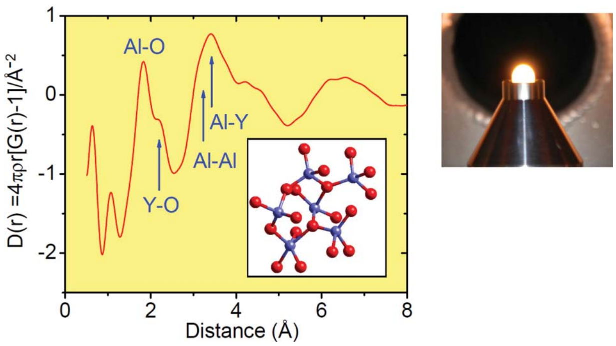

Figure 2: Total X-ray differential distribution function for $(Y_2O_3)_{20}(Al_2O_3)_{80}$ at 2200°C shown together with a group of tetrahedral AlO_4 oxygen triclusters (O:red, Al:blue). Insert: A high-temperature liquid droplet floating in an aerodynamic levitator.

of the High Pressure Synergetic Consortium (HPSynC). On the horizon of the APS Upgrade program, we also present an outlook for HP science at APS.

Highlights of High-pressure Research at APS

Noble element xenon becomes ignoble under pressure

Pressure can deeply modify intramolecular and intermolecular interactions, and be used as a novel reactive variable in chemical synthesis. As a result, novel chemical reactions and bonding constructs that are not observed under atmospheric conditions may emerge under extreme conditions, opening up intriguing possibilities for the discovery of new matter and novel methods of synthesis.

HP experiments at HPCAT at the APS revealed that the unreactive noble gas xenon readily reacts with molecular hydrogen under pressure while at room temperature. A novel compound $Xe(H_2)_7$—with the highest number of hydrogen molecules per chemical formula unit known to date—forms above about 4 GPa and surprisingly remains stable even up to 250 GPa. High-resolution structure study of this compound reveals that the xenon atoms coalesce into pairs that are surrounded by hydrogen molecules (Figure 1). Experimental data show the first evidence of the weak forces that "glue" these two elements together to form this solid. These results [5] have attracted the attention of theorists around the world. This research, in addition to opening up potential routes to synthesizing a new class of hydrogen storage materials, revolutionizes our understanding of xenon chemistry, with important implications in diverse fields such as energy research and planetary science. The answer to the "missing xenon paradox," which accounts for abnormally low xenon concentration in terrestrial atmospheres in comparison to that of the sun, may therefore lie in identifying similar xenon compounds formed at high pressure-temperature conditions.

Transitions in liquids

From a theoretical standpoint, the existence of a first-order liquid-liquid transition is entirely possible, so why don't we see them all the time? After all, polymorphic crystal-crystal transitions are commonplace. What is preventing us from fundamentally redrawing the liquid region of several seemingly blank phase diagrams? One part of the answer is that many of the proposed first-order *polyamorphic* liquid-liquid transitions occur in the difficult-to-access supercooled regions of phase space or at high pressures.

The liquid state is dynamic and inherently disordered, and can vary continuously with temperature and pressure, making subtle density and structural changes difficult to identify even on the world's best X-ray instruments. Accurately measuring an X-ray structure factor to within 1% over a wide momentum transfer range is difficult enough, but at extreme conditions this challenge is magnified. At APS sector 11 containerless levitation techniques (Figure 2) have been developed in tandem with the latest pair distribution function (PDF) instrumentation to probe metastable structures in supercooled fragile liquids. Such liquids tend to exhibit a dramatic rise in viscosity with decreasing temperature and are often highly metastable. The 11-ID-C high-energy X-ray station routinely operates at 115 keV and is home to an established levitation program using aerodynamic and acoustic techniques. At temperatures of ~1500°C polyamorphism was recently observed in a narrow compositional range of yttria-alumina melts [6].

Gradual transitions in glasses have also been observed in HP PDF measurements at sectors 1 and 11. These experiments use microfocusing optics and perforated diamond anvil cells (DAC) to minimize unwanted Compton scattering from the diamonds. The technique has been successfully applied to several tetrahedral and trigonal network glasses relevant to the geological and ceramics communities to pressures of >40 GPa.

Nuclear resonant spectroscopy under high pressure

With advances in crystal optics, IXS with meV resolution in the range of 10–30 keV became possible, which provided new tools at sectors 3, 30, and HPCAT of the APS for measuring thermodynamic, elastic, and magnetic properties of materials under HP. In particular, of great interest in the HP community are: measurement of phonon density of states and the subsequent derivation of Debye sound velocity, which distinguishes compression and shear wave velocity, and their temperature and pressure dependence; anisotropy of sound velocity; determination of mode Grüneisen constants; and measurement of phonon dispersion relations.

The use of Mössbauer nuclei as a probe of vibrational density of states has opened the possibility of characterizing opaque samples [7] like iron, iron-rich spinels, and perovskites. Since the technique is isotope selective via the Mössbauer nuclei, typical problems associated with sample environment leading to deteriorating signal-to-background ratio are readily resolved. By using focused X-ray beam and tunable high-resolution crystal monochromators with 1-meV resolution, partial phonon density of states is recorded for iron and a variety of iron alloys including hydrogen, carbon, oxygen, sulfur, silicon, and nickel

(Figure 3). Studies carried out up to 1700 K under pressure led to a re-evaluation of Birch's law [8] and spin changes in magnesiowustites.

Currently, APS beamlines offer five different isotopes for nuclear resonant inelastic X-ray scattering (NRIXS) and nuclear forward scattering (NFS) studies under HP, with compounds containing Kr, Fe, Eu, Sn, and Dy.

Earth and planetary sciences

Research at conditions relevant to the Earth and planetary interiors (Figure 4) is being performed at many beamlines at the APS. Among them, GSECARS is a dedicated sector with its main program focusing on geoscience. Equations of state, phase transformations, element partitioning, and electronic and optical properties of various materials have been successfully studied at extreme conditions.

Among the tools used for generating high-pressure and high-temperature conditions, the double-sided laser heated DAC technique is commonly used and installed at several APS beamlines [4,10-12] for various X-ray measurements. The large-volume press (LVP) is complimentary to the DAC, with large (millimeter-size) samples, better-characterized pressure, temperature environment, and the abilities to control non-hydrostatic stresses and strain. The LVP techniques are ideal for studying bulk properties of complex materials, which are frequently encountered in Earth science. There are two LVP systems at GSECARS, a 250-ton system at the bending magnet beamline and a 1000-ton system at the insertion device beamline [2]. Phase relations including melting curves of several important Earth materials have been determined by *in situ* phase identification using X-rays. By implementing acoustic transducers, both compressive (Vp) and shear wave (Vs) velocities of polycrystalline solids have been measured along with specific densities at simultaneous high P and T, to construct P-V-Vp-Vs-T equations of state. This has allowed construction of equations of state without relying on a specific pressure standard. The development of the deformation DIA (D-DIA) [13] has greatly simulated the studies of rheological properties of materials high P and T. Stress-strain curves have been measured at controlled strain rates to construct flow laws for olivine, ringwoodite, hcp iron, and others. Measurements on serpentinite have enabled evaluation of dynamic stress build-up in subducting slabs. A high-pressure tomography microscope (HPXTM) has been installed, which allows direct imaging of samples under high P and T by X-ray microtomography [14]). This new tool has opened a field of

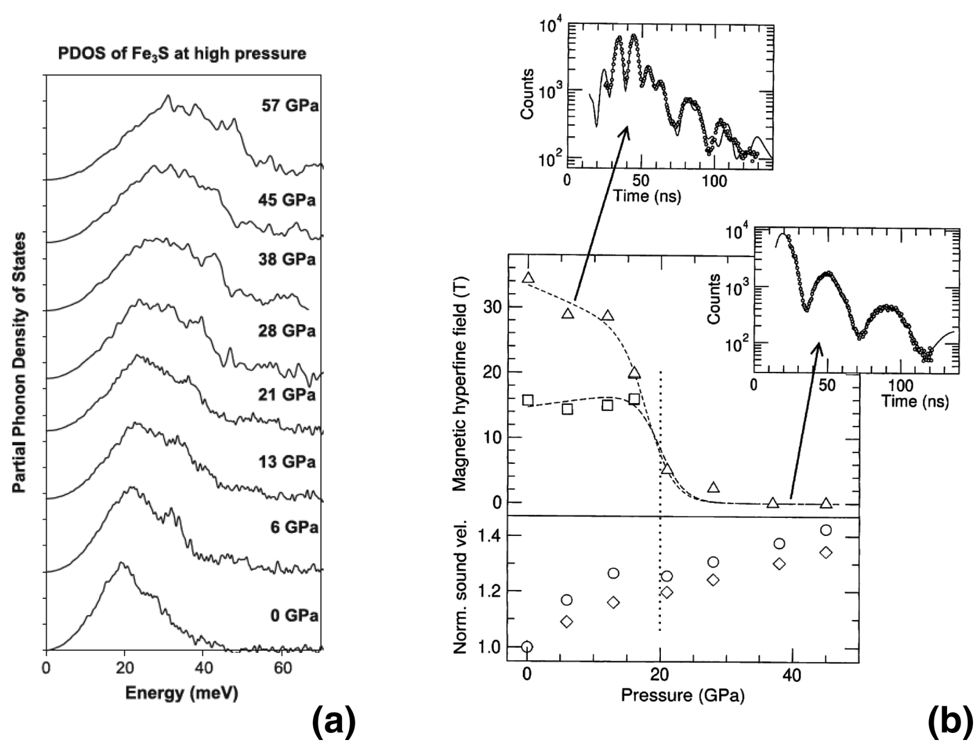

Figure 3: Collapse of magnetism measured by NFS and its impact on sound velocity in Fe_3S derived from NRIXS data: (a) partial phonon density of states; b) collapse of magnetism (after [9]).

high-pressure imaging and inspired studies on interfacial tension of melts, equations of state of glasses and melts, and fabric evolution of composite materials under large shear deformation.

High Pressure Synergetic Consortium (HPSynC) at the APS

A facility-wide approach to HP-SR research has been established at the APS through the High Pressure Synergetic Consortium (HPSynC), which is facilitated by scientists from the Carnegie Institution of Washington. Established in 2007, the goals of HPSynC are to establish HP environments at the APS, to make novel synchrotron techniques available to HP researchers, and to establish a gateway for HP scientists to many beamlines at the APS. Rather than concentrating on one beamline, HPSynC staffs conduct cutting-edge research and provide support on a range of instruments (such as high-P-T vessels and analytic probes) on many beamlines. In the process, they bridge scientific disciplines and communities.

HPSynC has been focusing on four areas: (1) developing novel HP-SR techniques; (2) coordinating existing equipment and tools; (3) establishing an advanced sample-preparation laboratory; and (4) creating a series of portable systems dedicated for HP research. Since 2007, successful HPSynC pilot projects have demonstrated the importance of the synergetic consortium between HP programs and state-of-the-art SR techniques. For example: (1) utilizing the X-ray nanobeam for ultra-high-pressure studies and for nano single-crystal diffraction; (2) integration of multiple synchrotron techniques—such as X-ray spectroscopy, micro-XRD, and NRS—for studying the phase transition mechanisms and dynamics of electrons, atoms, and spins; (3) development of nanoimaging techniques for HP research to study composite materials, the morphology of individual particles, plastic/elastic deformation, and the equation of state of non-crystalline materials at HP; and (4) close collaboration with theoretical calculation scientists for detail modeling and to control and predict materials properties at extreme conditions.

Outlook of HP Research at APS

HP research is a vibrant field and one of the main science drivers for the APS Upgrade program on the horizon. In the current roadmap planning, long straight sections of up to 8 meters will be installed at both HPCAT and GSECARS, which will offer a significant increase in capacity at these dedicated HP beamlines. The brighter source will enable nanoprobes to be developed, higher energy resolution in IXS, and finer time resolution in time-resolved experiments. Ever more complex samples at increasingly broader pressures and more extreme temperatures will be studied with higher-accuracy probes for characterization of structural, electron, and phonon properties.

Research activities under extreme conditions will continue to grow at many other APS beamlines. The nano-XRD probe at sector 34 will be equipped with X-ray optics with a working distance suitable for a variety of DACs together with ultra-stable sample stages and advanced detectors for studying materials at even more extreme conditions (toward 1 TPa) or less homogeneous samples. Precise single-crystal studies of HP phases can be performed with the nano beam for individual crystallites within a power sample. HP PDF studies will greatly benefit from developments in micro-focusing optics and the advent of faster and more efficient area flat plate detectors. A move towards energy discriminating large area detectors would further increase in the quality of collecting high-Q structure factor data. At sectors 2, 32, and 34, imaging capabilities (coherent diffraction imaging, mass density tomography, full field imaging) are being actively integrated for HP studies with resolutions from 1 μm down to 30 nm, which will open a new era of

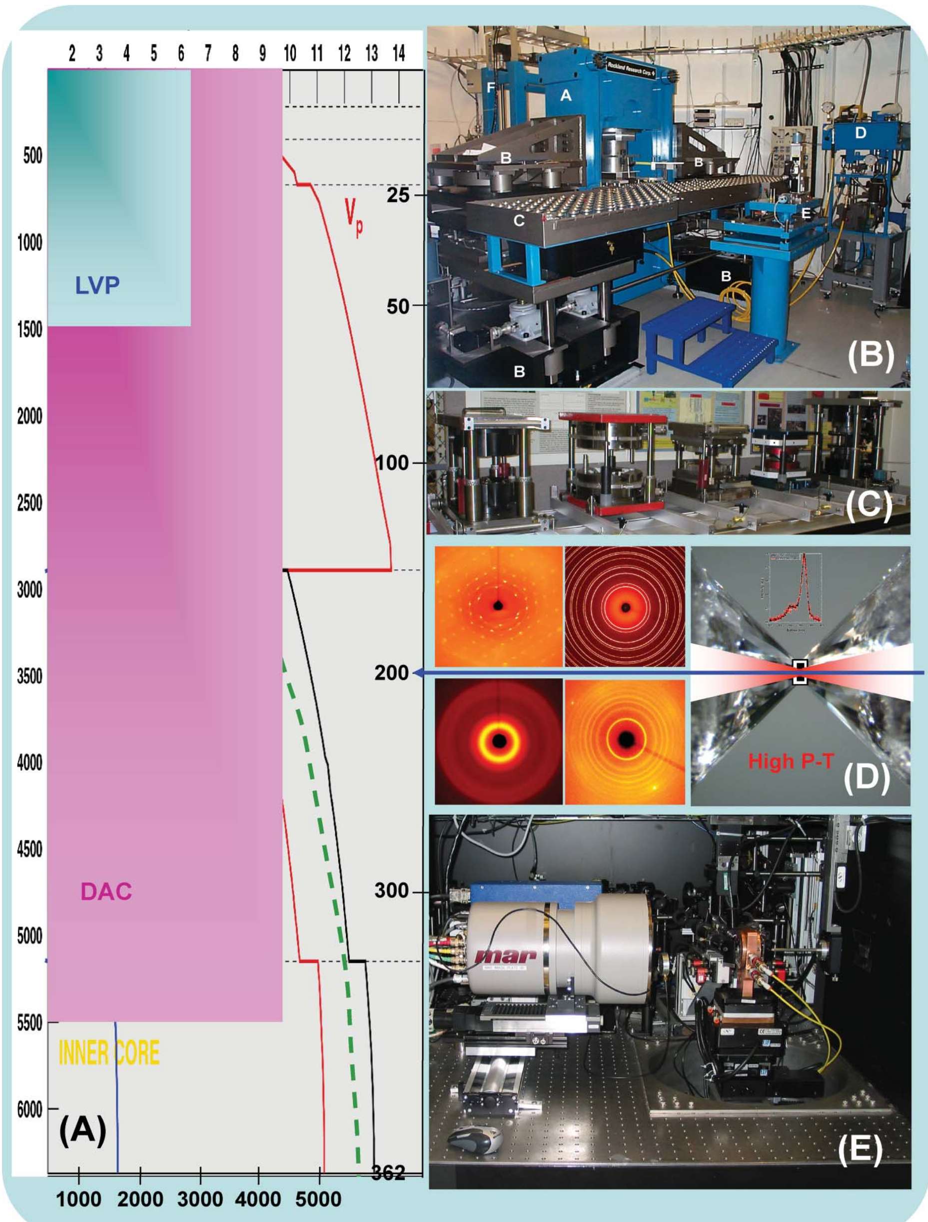

Figure 4: (A) Structure of the Earth according to seismic data. Vertical axes are depth (km, left) and pressure (GPa, right). The top horizontal axis is for compressional (Vp: red) and shear wave (Vs: blue) velocities (km/s), density in g/cc, and the bottom horizontal axis is temperature (°C). The pinkish and bluish boxes depict current P-T capabilities of the DAC and LVP, respectively. (B) The 1000-ton LVP system at 13-ID-D. (C) Various devices used in LVP system. (D) The double-sided laser-heated diamond anvil cell. (E) Experimental setup for XRD with the advanced flat-top double-sided laser heating system at 13-ID-D.

imaging HP phases, for studying the plasticity, elasticity, and precise equations of state of amorphous/liquid materials. The HP NRS techniques are particularly flux limited and will greatly benefit from the higher flux with the APS Upgrade. The increased flux will allow for developing NRS techniques with higher energy resolution, and enable many new *in situ* experiments for more detailed information on electronic excitations and plasmons, and finer features in x-ray Raman. Because HP samples are always embedded in surrounding materials (anvils, gaskets), excellent collimation is required to remove signals from surrounding materials. Developments for precise collimation down to micron size for various IXS techniques are being carried out by APS scientists, which will effectively increase the signal-to-noise ratios. A beamline dedicated to dynamics compression science is proposed for time-resolved studies in dynamic processes and would be the first of its kind at a third-generation high-energy synchrotron facility. Dynamic compression capabilities (utilizing high-velocity impacts, high-intensity lasers, and pulsed power generators), coupled with ultrafast measurements facilitated by the APS Upgrade, represent the most versatile approach for achieving the widest range of thermo-mechanical conditions in a controlled manner.

Supporting facilities for HP research and sample preparation laboratories are the important component in the APS Upgrade. For instance, a micro-machine for crystal cutting and sample shaping is proposed. A large D-DIA with 30-mm truncation on the first-stage anvils has been installed at the insertion device beamline and is currently being commissioned, pushing pressure conditions towards the megabar (100 GPa) at high temperature with LVP. Various new types of DACs are being developed for ultra-high pressure, large opening, or compactness to fit in the varieties of X-ray measurements. More gas loading systems will be developed for various gas types (including H_2) and for loading gases cryogenically. Portable systems, including laser heating systems, optical Raman and ruby fluorescence systems, and microfocusing mirrors, have been developed and will be integrated at many APS beamlines. In addition, working with the detector group at the APS, advanced detectors are proposed for HP experiments. Together with the source upgrade and developments in HP-SR techniques, these supporting facilities will serve as a strong base for the next level of scientific and technical integration for a very bright future of HP science at the APS.

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