Materials at High Pressure

Future directions in high pressure science and instrumentation at NSLS & NSLS-II

From the breakout session
“Materials at High Pressure”

during the joint workshop for: “The NSLS-II Powder Diffraction Project Beamline” and “Materials Science Engineering Strategic Planning for NSLS and NSLS-II”

January 17-18, 2008

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This report is prepared from the results of the breakout session and by using the white papers: ***“High Pressure Needs at the NSLS II Synchrotron”*** from the NSLS II workshop July 17 – 18, 2007 by *Donald Weidner* and *“**Earth Science Research Perspective (Material Properties at Extreme Conditions) at the NSLS II” by** Jiuhua Chen, Malcolm Nicol, Donald Weidner, David Mao.*

Introduction

Pressure is one of the fundamental thermodynamic variables, which can be varied over a range of more than sixty orders of magnitude, from the vacuum of outer space to pressures in the interior of neutron stars. The exploration of matter at extreme conditions is a central theme in a broad range of scientific disciplines (e.g. material science chemistry, physics, and Earth and planetary science). The application of pressure can induce both continuous and discontinuous changes in atomic and electronic structure. Learning how atomic and electronic arrangements change under extreme conditions provide insight into the nature of phase transformations, chemical reaction, and also evolution in micro- and nanostructural components, such as crystallite size, dislocations, voids, and grain boundaries. Once these processes are understood, it will be possible to predict responses of materials under thermomechanical extremes using advanced computational tools. Further, this fundamental knowledge will open new avenues for designing and synthesizing materials with unique properties. Using these thermomechanical extremes will allow tuning the atomic structure and the very nature of chemical bonds to produce revolutionary new materials.

Powder and single-crystal diffraction and infrared spectroscopy have been on the forefront of synchrotron-based techniques used to study materials at high pressure. Many scientific breakthroughs have been made during the last 30 years that advanced our knowledge of materials at high pressure and the structure of Earth's interior. The introduction of 3rd generation synchrotron sources about a decade ago has lead to a major step forward in high-pressure science. New applications of synchrotron spectroscopy methods at high pressures in a diamond anvil cell (DAC) have been developed at the third-generation synchrotron sources: spin-sensitive x-ray emission, nuclear resonant scattering methods, inelastic scattering from electron and phonon excitations, resonant inelastic scattering techniques (RIXS). The rapid development of inelastic scattering techniques provides a multitude of probes of elementary excitations in condensed matter in a broad energy and momentum parameter space. As a result of synergetic developments in synchrotron and pressure cell design a broad range of x-ray studies of the physical and chemical properties of solids can be now conducted *in situ* at high pressures to several hundred gigapascals.

Addressing forefront scientific questions in high-pressure research is only achievable through combination of the results from experiments obtained with complementary high-pressure techniques. Furthermore, current scientific challenges in high pressure involve multiple extreme conditions in addition to the static or dynamic high pressure environment, e.g. high and low temperature, magnetic fields. The combination of state-of-the-art diffraction with optical and X-ray spectroscopy, imaging and computational methods open new frontiers in the study of the high pressure behavior of materials.

Scientific Challenges

The scientific challenges in high-pressure research are manifold and involve overlapping scientific disciplines. In the past decade, advances in synchrotron x-ray based analytical techniques fostered remarkable breakthroughs in high pressure sciences, including discovery of the post-perovskite phase of $MgSiO_3$ stable at more than 100 GPa (2, 3), the unusually low melting temperatures of sodium, reaching 300 K at 118 GPa (4), complex structures of alkali metals at high pressure (5-8), observation of a spin state transition in iron under conditions in Earth's lower mantle (9), the changes in the bond characteristic of compressed graphite (10), and the measurement of the spin and charge order in chromium at high pressure (11).

The following chapter describes the current and future scientific challenges defined by the high-pressure community at workshops and conferences held during the period of 2005-2008. The scientific case covers challenges from several fields, such as material science, chemistry, physics, and earth sciences. Some classifications for these scientific challenges are arbitrary, since they would fit in any of the scientific fields mentioned above.

Earth and Planetary Science

Study of the structure, composition, and history of the Earth and planetary interiors is an extremely challenging task because the deep interior is inaccessible. Seismological investigations are the primary source about the interior structure of the Earth and other planets. Advances in seismology provide detailed 3D tomographic images that show heterogeneities, anisotropy, attenuation and discontinuities from the surface to the center of the core. Seismic models of the variation of compressional and shear wave velocities and densities presumably reflect radial and lateral variations of chemical composition, mineralogy, pressure and temperature. Experimentally derived information on the density and elastic properties of Earth and planetary materials under geologically relevant pressure and temperature conditions are needed for successful interpretation of the seismic models.

Refinement of the Earth's interior structure

Subduction Zones

Subduction zones (Figure 1) are the geologically most active areas on Earth, greatly impacting life on Earth's surface (12-15). Subduction zones exist at convergent plate boundaries where oceanic lithosphere converges with another plate and plunges into Earth's interior. Thereby, crustal and upper mantle materials get recycled into the mantle.

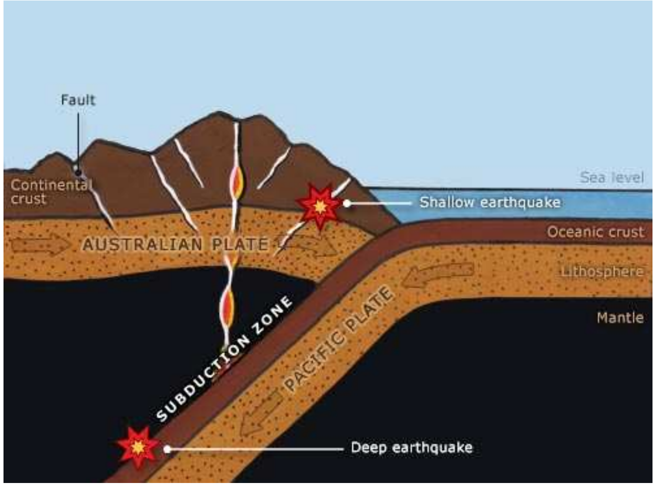

Figure 1 Structure of a subduction zone. The descending plate presses against the overlying continental plate, causing the overlying plate to fracture and producing shallow earthquakes. Deep earthquakes occur in the oceanic crust that is being bent downward into the subsurface. As the Plate descends into the hot interior of the earth, rock near the plate boundary starts to melt, feeding volcanoes above.

Increased volcanic and earthquake activity can be found at plate boundaries, where subduction is active. The general mechanism of subduction is well understood through the concept of plate tectonics. However, the processes in the subduction zone, that lead to volcanism and earthquakes are poorly understood. The technical capabilities to study the complex processes in subduction zones *in situ*, have recently emerged with the availability of 3rd generation synchrotron sources. With the current experimental facilities at hand and the unique experimental capabilities that NSLS-II will provide on the horizon, experiments that simulate the complex conditions (pressure, temperature, composition, stress etc.) in the subduction zone are in reach.

One of the key issues related to subduction zones is the influence of volatiles, in particular water and carbon dioxide, on mineral and rock properties and stabilities at high pressure and temperature. The determination of the efficiency of the recycling of volatiles in the mantle and the identification of possible mineral reservoirs for volatiles at Earth's mantle conditions will allow us to gain a deeper understanding of Earth's water and carbon dioxide budget and Earth-Atmosphere interactions. Synchrotron IR spectroscopy at extreme conditions is the only technique that allows *in situ* investigations of the volatile content of minerals and provides information on the speciation of hydrous components. In the lower mantle, where silicate perovskite (PV) and magnesiowüstite (MW) are the stable assemblage, the water storage capacity remains uncertain, with current estimates ranging by three orders of magnitude, from ~1 ppm (16) to over 2000 ppm wt. H_2O (17) in silicate perovskite. The major question now is whether or not hydrogen observed in synthetic samples occurs as structurally bound hydroxyl in the perovskite, or is present as hydrous mineral inclusions or melt quench.

Earthquakes are among the most destructive natural events happening in subduction zones and are almost impossible to predict. The earthquake process involves the interaction of stress fields with minerals and volatiles from the macro scale to the micro scale. Deep earthquakes occuring along subduction slabs offer a natural probe of the Earth's interior. However, the mechanisms of these earthquakes have remained uncertain for decades. Improved understanding of these dynamic phenomena requires *in situ*, time-resolved studies of surface physics and chemistry during stress and strain episodes. High-resolution strain mapping, chemical mapping, and interface phenomena at controlled environmental conditions of pressure, temperature, and stress holds the hope of new insights into the phenomena of earthquake processes.

Core-Mantle Boundary

The Core-Mantle-Boundary (CMB) at depth of 2900 km separates the liquid outer core from the silicate lower mantle (Figure 2). The change in physical properties across this boundary is as great as that between the solid Earth and the atmosphere at Earth's surface. The CMB region is highly anomalous, and holds the key to a number of fundamental geophysical questions including: thermal structure of the deep Earth, origin of geochemical heterogeneity, ultimate repository of subduction slab material, and chemical interactions between the mantle and core. Just above the CMB at 2700 km a discontinuity in seismic wave velocities is observed commonly referred to as D”. Recent advances in high-pressure science make it now possible to routinely generate pressure and temperature conditions of the CMB in the laboratory.

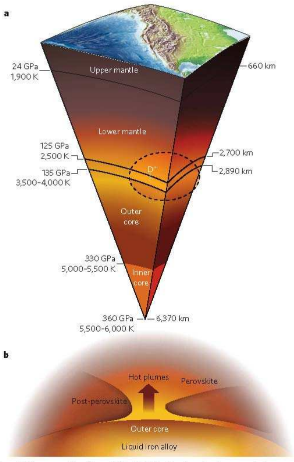

Figure 2 Cross section of Earth's interior. B. Close up showing possible complex structures near the core mantel boundary (from (1)).

The discovery of the structural phase transition from perovskite to $CaIrO_3$-type phase (post-perovskite) (2) at pressure and temperature conditions of the D” discontinuity has revolutionized our understanding of the CMB (2, 3) (Figure 3). But many fundamental questions regarding D” and the CMB remain (18). The interaction of the molten outer core with the silicate mantle is largely unknown (19, 20). Knowledge of exchange reactions at high pressures and high temperatures between a metal from one side and refractory oxides and silicates from another side is important for understanding the early Earth differentiation. The temperature of the liquid outer core and the temperature profile over the CMB are still unknown (21). Measurements of the melting temperature, elastic properties and crystal structures of iron alloys, metal oxides and lower mantle silicates (e.g. perovskite and post-perovskite) at high pressure and temperature will allow

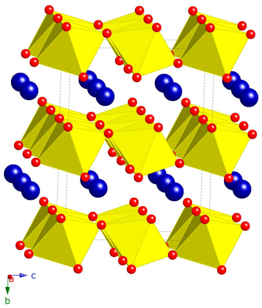

Figure 3 Crystal structure of $CaIrO_3$-type (post-perovskite) phase of (Mg,Fe)SiO3 (from (1)).

us a further insight into the composition, temperature, and structure of the CMB.

Mission to the Earth's core and beyond

The Lehmann discontinuity at 5150 km marks the boundary of the liquid outer core and the solid inner core. The structure and properties of a planetary core promise to provide a wealth of information about the evolution and dynamic processes within the planet. However, limitations in pressure generation and related properties measurements at such extremely high pressures and temperatures hinder the mission to the Earth's core (21). As analytical techniques set restrictions on the minimum sizes of high pressure samples and higher pressures normally require smaller samples, the sub-micrometer spatial resolution of the NSLS II X-ray beams will enable new ground breaking work to challenge accessibility to the pressure at the center of the Earth's core (3.5 million atmospheres) and beyond. The high spatial resolution X-ray beam will also significantly enhance the capability to recognize heterogeneities of pressure, temperature, chemistry, structure and phase within small samples and to improve reliability of experimental data obtain at such high pressures. With such advances in experimental capabilities, we expect to address the long-standing issue of core composition and to understand the new observations of inner core seismic anisotropy, super-rotation and magnetism.

Rheology

The plastic properties of ceramics – rocks – at high pressure and temperature control the evolution of the Earth and planets (22). The pressure envelope for quantitative rheological experiments has limited us to properties of the upper layers of the Earth until very recently when synchrotron methods have extended the pressure range by about two orders of magnitude. This has come with synchrotron X-ray imaging techniques and stress metrics. The current experimental capability limits the strain precision to 10_{-4} (resolving 100nm length change over 1 mm long sample) in multi-anvil apparatus. The high spatial resolution of less than 10 nm will improve by one to two orders of magnitude strain precision to about 10^{-6} and strain rate precision to 10^{-9}-10^{-10} s^{-1} and will significantly reduce the gap of strain rates between laboratory experiments and geological flow (10^{-12}-10^{-16} s^{-1}). On the other hand, seismic studies use millihertz acoustic waves to probe the Earth, while laboratory studies often use megahertz acoustic waves. Differences in these time scales are expressed in the attenuation, or the Q (quality factor), of stress-strain relationships. To measure Q, we need to measure stress relaxation times and strain retardation times as a function of frequency. With the advances of synchrotron tools, unprecedented flexibility in controlling the stress and strain during the deformation process at high pressure and temperature is feasible. The strain precision to be achieved at the NSLS II will allow us to describe seismic wave attenuation and related transient creep with a requirement of strain resolution (10^{-6}) relevant to seismic attenuation in the mantle. Finally, the high spatial resolution could enable quantitative flow measurements in a diamond anvil cell. This will be a new era of rheology research, promising to extend the pressure envelope for rheological studies nearly one order of magnitude, from the pressure limit in multi-anvil apparatus to that of diamond anvil cell at the same strain precision of 10^{-4} (resolving less than 5 nm length change over 10 μm long sample). Knowledge gained thereby will place important constraints on the thermal, velocity, and density structure of Earth's interior.

Planets

The discovery of 273 extra solar planets, since 1995, demonstrated that planetary systems are very common in the universe. Understanding the formation, evolution and current structure and dynamics of planets is a paramount challenge for planetary scientists. High pressure and temperature experiments can help interpret the data gained from satellites and exploration missions on the current structure and dynamics of planets and provide insight into planetary evolution. The scientific challenges differ from the ones in Earth sciences, since the range of compositions and thermodynamic conditions in the solar system is much greater than for Earth. The 8 planets and the planetary bodies in our solar system can be divided in four categories: Gas giants (Jupiter, Saturn), Ice giants (Neptune, Uranus), ice/rock bodies (Europa, Ganymede, Callisto, Titan, Triton, Pluto), and terrestrial bodies (Mercury, Earth, Mars, Venus, Moon, Io).

The investigations of hydrogen and hydrogen-helium mixtures at high pressure and temperature are key to understand the structure of gas giants and will contribute to understanding their formation. Precise measurements of the equation of state of hydrogen and hydrogen-helium mixtures in the relevant pressure and temperature range will help to determine if Jupiter has a rocky core. Furthermore, the detection of continuous or discontinuous transitions in the high pressure and temperature behavior of H_2 and $He\text{-}H_2$ mixtures will provide important information about the interior structure of gas giant planets (23).

High pressure and temperature investigation of water, water mixtures with ammonia, methane, and water-rock interaction will provide further insight into the interior structure of ice giants and solid ice/rock bodies (24, 25). The determination of the properties of aqueous fluids at moderate pressure and temperature, e.g. reactivity, although technically very challenging, is crucial to understand the internal evolution of many solid ice/rock bodies in our solar system.

Further insight into the formation, structure and evolution of terrestrial planets can be gained by determining the phase diagrams of chemical compositions relevant to these planets experimentally and correlate these data with results from exploratory and satellite missions. Extra solar terrestrial planets which encompass up to 10 Earth masses are expected to have central pressures and temperatures up to 3500 GPa and 8000 K, much greater than found in terrestrial planets in our own solar system (26) and constraining their interior structure will consequently require experiments to extend to much more extreme conditions.

Material Science and Chemistry

Super hard materials

Compounds can be defined as super hard materials, when their micro-hardness exceeds 40 GPa. In addition to high hardness, they usually possess other unique properties such as compression strength, shear resistance, large bulk moduli, high melting temperatures, chemical inertness, high thermal conductivity, etc., which makes them materials highly desirable for a number of industrial applications. One prominent goal is to synthesize new phases in systems such as B-C-N-O or Si-B-C-N that are thermally and chemically more stable than diamond, and harder than cubic BN, and thus would be excellent materials for high-speed cutting and polishing of ferrous alloys. The most common conditions employed to synthesize super hard materials involve extreme pressures or extreme pressures in combination with temperatures. Fundamental research to better understand the atomic structure and the bonding characteristics are badly needed. Knowledge of these fundamentals will help materials scientists understanding how and why a material becomes super-hard.

The source characteristics of NSLS-II and the unique experimental capabilities at the proposed high-pressure stations will open new possibilities in synthesis and characterization of super hard materials.

Nano-crystalline Materials

The field of nano-crystalline materials is in rapid development (27). Materials consisting of nanometer-sized crystallites are characterized by a large fraction of surface or inter-surface atoms. Correspondingly, these materials have novel physical and chemical properties compared to their bulk counterparts, which could lead to new functional materials. One of the main goals of high pressure research in this area would be a thorough investigation of crystallite size effects on the equation of state (bulk modulus and other elastic constants), strength (28) and possible solid-solid phase transformations. As for nano-materials, the common rule seems to be that the smaller the particle size, the higher the transition pressure. However, a few systems with the opposite trend have been reported in the literature. Competing processes are determining the high-pressure behavior, and there is a need for systematic studies of the influence of crystallite size on the transition pressures and other physical parameters.

Recent developments in high-energy X-ray total scattering techniques at high pressure combined with pair distribution function analysis allow now extracting information of the atomic structure of nano-particles from diffraction data. This new technique will mature over the next years and will provide fundamental insight in structure-property relationships of nano-crystalline materials at extreme conditions.

Porous Materials form Micro- to Nano-Pores

Porous materials play an important role in many technological processing applications (29). These materials are widely used as catalysts, catalyst supports and membranes, and form the basis of new technologies, involving energy storage, novel reactions, waste sequestration etc. Any process that takes place within the pores of a solid is strongly influenced by the geometry and topology of the host's pore matrix. Therefore, the determination of the structural or physicochemical parameters that are related to the mechanism of these processes is the key for the characterization of porous materials. In order to understand the processes in the pores, the bonding and dynamic of guest ions and molecules in the porous compounds need to be determined (30, 31).

The application of pressure to porous materials often leads to unexpected structural response, e.g. amorphization (32), penetration of liquid media into the pore volume (30). Understanding the mechanism of geometric deformation of the structure with pressure and temperature is important to evaluate its stability at non-ambient conditions. Compressibility and thermal expansion data are necessary for thermodynamic calculations and to yield thermo-chemical parameters with internal consistency. Possible interactions with penetrating pressure transmitting media will give new insight on the nanoscopic poroelasticity of the materials (30). The thermal behavior of many porous materials has been extensively studied and interesting effects, for example negative thermal expansion behavior have been discovered (33). However, the literature on the effect of pressure is limited, and until recently little quantitative information on pressure-induced structural modifications and phase transformations has been reported. The combination of structural data of the host and guest structure and data on the dynamic and mobility of the guest yields the information necessary to clarify the mechanism of structural deformation and to understand technological relevant processes.

Chemical Reactions and Reaction Kinetics

Chemical reactions are ubiquitous in nature. Their comprehensive studies are necessary for defense, industry, and academic science. Better understanding of the chemical reactions in the condensed phase at elevated pressure and temperature is currently required. Almost all industrial and synthetic chemistry occurs in the condensed phase. Chemistry in the condensed phase is the basis of life. Condensed phase chemistry is essential to planetary processes on the Earth as well as other terrestrial bodies. The knowledge of the reaction chemistry of energetic materials at high pressure and temperature is necessary to understand their behavior under impact and in detonation conditions. Understanding of a nature of chemical reactions in simple molecular materials (e.g., hydrogen and nitrogen) will provide a basis for designing and synthesizing of fuel for the future.

Following chemical reactions using the unique time structure of synchrotron sources will allow deep insight into reaction kinetics and reaction mechanisms previously inaccessible. The high brilliance of NSLS-II will facilitate these *in situ* studies of reactions at extreme conditions.

Synthesis of Novel Materials

Research at elevated pressure and temperature provide a new insight into synthesis of materials with properties important for industrial, technical and scientific applications (34). These include super hard materials, high-temperature superconductors, ferroelectrics, multiferroics, high energy density materials, hydrogen storage materials, materials for computers and communications, and nano-materials. High-pressure studies provide otherwise unattainable information about the phase diagrams, thermodynamic properties, and electronic structure which can predict directions for search of materials with desirable properties. Moreover, high-pressure synthesis remains unique in many cases.

Physics

Element Structure and Complex Alloys

The exploration of high-pressure modifications of elements has revealed a surprising complexity of their properties and crystal structures. Recently, a rich polymorphism under high pressure in elementary materials has been observed and reported. In addition, theoretical studies predict highly unusual properties of materials containing light elements, e.g. superionicity, metallization and superconducting superfluids.

Light elements like hydrogen, sodium and lithium are a long-standing subject of scientific interest. For hydrogen, predicted of metallization motivated a large scientific effort over many decades. For lithium, the recent discovery of low-symmetry high-pressure allotropes in combination with theoretical findings concerning the compression-induced transition of the valence electron from s- to p-like behavior has stimulated a number of investigations concerning their physical properties (35-37). Measurements of the melting curve of sodium revealed an unexpected and unpredicted behavior at high pressures. The melting temperature of sodium reaches a maximum at about 31 GPa and shows a decrease in the melting temperature to 300 K at 118 GPa (4). The experimental exploration of light elements at high pressures requires the brilliance of 3rd generation synchrotron radiation sources, due to their weak scattering power.

A large number of elements show unexpected electronic transformations e.g. metal-insulator transitions, or transition to superconducting state (Figure 4).

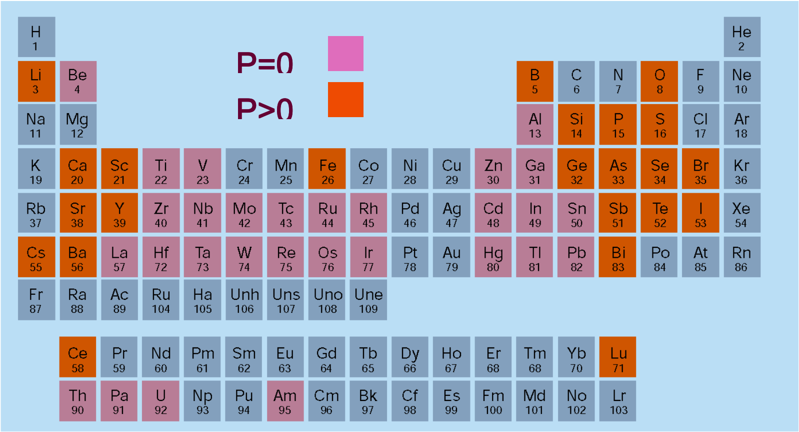

Figure 4 Periodic table of the elements. The purple color highlights the elements for which superconductivity was observed at high pressure (Figure from Ashcroft (37)).

It was shown that several elements, e.g., the heavy alkaline metals undergo pressure-induced s-d transitions. In the regime of the electronic change the occurrence of low symmetry atomic arrangements has been observed. Some of the structural patterns correspond to partial structures of intermetallic compounds, e.g., that of Rb-IV (38) (Figure 5) to that of W$_5$Si$_3$.

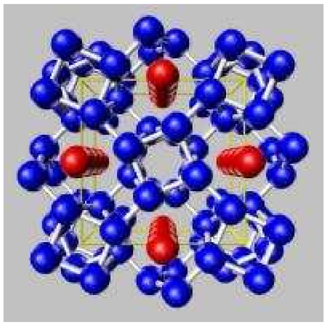

Figure 5 The host-guest structure of Rb-IV showing host (blue) and guest (red) atoms. (Figure from McMahon *et al* (38))

Similar framework structures have been found for the allotropes Bi-III and Sb-II, which are additionally characterized by incommensurate modulations of host and guest lattice. In the light of these investigations, phase stability and crystal structures of a number of as-yet undetermined high-pressure modifications of elements like silicon and germanium have been characterized. The studies of metallic elements and the findings concerning their electronic and structural organization have motivated a number of projects investigating the pressure dependence of the electronic configuration in intermetallic phases. It was shown that in a number of ytterbium containing intermetallic compounds compression induces changes of the oxidation state of the rare-earth metal. The observations of these valence transitions have become a focal point of ongoing theoretical and experimental investigations.

Highly Correlated Electron Systems

Systems with strongly correlated electrons have fascinated materials scientists time and again, by revealing such intriguing phenomena as high temperature superconductivity, the colossal magneto resistance, and heavy fermion behavior and unusual dynamic ground states. There is growing evidence that in many of these systems, as for example the transition metal oxides, the unusual physical properties result from a competition between different electronic phases, of which some show instability towards a nanoscopically inhomogeneous electronic ground state (39). Understanding the phenomenon of “competing-order” and the inherent electronic inhomogeneities is considered to be key to identify the microscopic mechanism of many of these exotic ground states. In principle, pressure provides a formidable tool to study these phases, as it allows manipulating the band structure as well as the lattice parameters and symmetry. However, the challenge has been to detect the ultra weak lattice modulations associated with the electronic in homogeneities in a high pressure environment, and only recently groups have succeeded (11). The NSLS-II will enable such diffraction experiments with an unprecedented degree of resolution, and provide a direct probe of electron-lattice interactions in some of the most unconventional states of condensed matter at high pressure.

The ability to combine high pressure with other extreme environments such as high magnetic or electric fields is vital for studies of complex interactions in strongly correlated electron systems, such as questions concerning the competition or coexistence of magnetism and superconductivity, or the interplay between electronic order and Fermi surface instabilities. The combination of high pressure with other extreme environments (in addition to temperature) is world wide still in its infancy. The NSLS-II offers a unique chance to develop a world-leading program.

High Pressure Program at NSLS

High-pressure research has a strong presence at NSLS. Currently, four experimental end stations are dedicated to high-pressure research using diamond anvil cells and large volume presses. The experiments are located at the superconducting wiggler X17 at the X-ray ring and the bending magnet U2 at the UV ring. X17B2 provides the high-pressure community with state of the art capabilities for experiments in large volume presses. Diffraction experiments at high pressure and temperature in a diamond anvil cell can be performed at X17B3 and X17C. The bending magnet beamline U2A offers the unique capability of conducting infrared spectroscopy measurements at high pressure and moderate temperatures using a diamond anvil cell.

The experimental capabilities provided by the high-pressure program at NSLS serves each year a user community of about 200 users from 50 national and international institutions. The topics of the conducted experiments originate in a range of scientific disciplines, such as material sciences, physics, Earth sciences and chemistry.

During the past six years, the high-pressure program a NSLS has been mainly supported by NSF, through COMPRES (COnsortium for Materials Properties Research in Earth Sciences). Additional funds for the IR spectroscopy beamline U2A have been provided by DOE CDAC (Carnegie/DOE Alliance Center) and by NSF-EAR and DOD for large volume program at X17B2.

The high-pressure program at NSLS is currently improving the capabilities of the experimental stations by addition of new hardware and by development and implementation of new experimental techniques. Some of the new additions are already made with the unique beam characteristic of NSLS-II in mind.

The following additions will be made to the beamlines during 2008 and 2009:

- **X17B2**
  - Development of a monochromatic X-ray diffraction side station. The side station will allow experiments in a Paris-Edinburgh type pressure cell at pressures up to 30 GPa and about 2000 K. Experiments in side station can be conducted simultaneously with experiments in the large volume press resulting in more effective use of beamtime.
  - Implementation of a new ten element solid state detector for precise stress measurements.

- **X17B3**
  - Development of a compact laser heating system based on a Yb:fiber laser. This will add simultaneous high pressure and temperature capabilities to the beamline. Furthermore, this is the first step towards the development of a potentially portable laser heating system for NSLS-II.
  - Development of micro beam capabilities (beam size ~ 1μm) by focusing with kinoform refractive lenses in collaboration with K. Evans-Lutterodt (NSLS). This development is the first step towards sub micron beams at the high pressure stations at NSLS-II (e.g. proposed Station A)
  - Further development of the total X-ray scattering technique at high pressures and temperatures for the investigation of disordered, amorphous and liquid materials in diamond anvil cells.

- **X17C**
    - Implementation of angle dispersive and energy dispersive single crystal diffraction, in collaboration with P. Dera (GSECARS).

- **U2A**
    - Development of in-situ high pressure and temperature synchrotron IR spectroscopy and applications to Earth sciences and material sciences under extreme condition (up to 300 GPa and 5000 K). The first step is to build an offline laser heating system based on a CO_2 laser, allowing the investigations of samples quenched from high temperatures.
    - Coupling dynamic-compression with pulsed synchrotron radiation for time resolved measurements. A feasibility study by a team led by Daniel Dolan from Sandia National Laboratories in October 2006 achieved an important milestone when they were able to couple a broadband synchrotron IR radiation from U2A beamline to characterize the emissivity of a copper film under shock compression.
    - Development of an experimental side station. The new facility will allow measurements on high-pressure samples with the highest spatial resolution possible at a synchrotron source while also having the highest broadband IR brightness. With a new microscope coupling a newly developed IR focal plane array detector and FTIR instrument, the facility will be ideal for mapping of natural samples (e.g., solid and fluid inclusions in thin section), heterogeneous charges from high-pressure experiments, as well as samples *in situ* at very high pressure in diamond or moissanite anvil cells.

Due to the continual development of the experimental stations, we expect a significant growth of the user community over the next years.

A large portion of the high-pressure research at NSLS takes place in experimental stations at the superconducting wiggler X17 and therefore depends on greatly on the reliability of this insertion device. The high-pressure community follows with great interest the solutions to the problem with the cryogenic cooling system of the wiggler X17. From the currently discussed solutions, the high pressure community is largely in favor of the replacement of the wiggler X17 with a new superconducting wiggler, which can be operated at reduced capabilities (limited field and/or periods) at NSLS and be transferred to NSLS-II. The advantages of this solution for the high pressure program at NSLS as follows: (i) the high pressure experiments would be served by a new and reliable insertion device; (ii) the reduced capabilities of the new superconducting wiggler would be equal or superior to the current wiggler; and (iii) a superconducting wiggler, suitable for high pressure research and taking full advantage of the unique source characteristics of NSLS-II would be present at the new ring on day one.

The high-pressure community applauds the decision of the NSLS to build a new beamline, X17A, at the superconducting wiggler port. The unique capability of this beamline will be ideal for investigation of disordered, nano-crystalline and amorphous materials at high pressure, using X-ray total scattering in conjunction with pair distribution function analysis. The condensed matter physics department at BNL has submitted an energy-related Laboratory Directed Research and Development proposal (Tranquada, Huecker, Bozovic, Davis). As part of this proposal it is planned to develop the capability to perform high-pressure single-crystal X-ray diffraction at low temperatures at X17A. This is in addition to the upgrade of this beamline for powder diffraction. The decision on this proposal is still pending. These additional high-pressure capabilities will further strengthen the high pressure research program at NSLS and could be transferred to a high-pressure or high-energy beamline at the NSLS-II.

The development of a new generation of X-ray detectors, lead by D.P. Siddons at NSLS, is of great interest to the high-pressure researchers. The current generation of area detectors was developed mostly for protein crystallography and therefore has the highest sensitivity at X-ray energies of 8-12 keV. Diffraction experiments at high pressure are usually conducted at energies of 30-40 keV, where these detectors have a remaining efficiency of less than 40 %. The proposed microstrip and a hybrid pixel-array detector using germanium sensors will be an ideal detector for diffraction experiments at high energies. High pressure diffraction experiments will mainly benefit form the superior signal-to-noise ratio, the fast read-out time and the large dynamic range. We envision the germanium hybrid pixel-array detector as the standard detector at the dedicated high pressure diffraction stations proposed for NSLS-II.

High Pressure Program at NSLS-II

The future scientific challenges in high pressure research involve sophisticated experiments on increasingly complex systems at ever higher pressures and temperatures. Most of the modern scientific questions in research at extreme conditions require integration of multiple experimental techniques. The sample environment for experiments at extreme conditions poses a rigorous restriction to sample size and volume and often contaminates the collected data. Therefore, the requirements on focal size, collimation and beam stability are very high. The small source size and the high brilliance over a large range of X-ray energies of NSLS-II will greatly benefit experiments at extreme conditions and stimulate new directions in research of materials at extreme conditions.

The high-pressure program at NSLS-II should have two components: dedicated high pressure beam lines and support for high-pressure research at a variety of other beamlines around the ring. Many high pressure cells are compact and portable and thus there is a prime opportunity to integrate high-pressure techniques into other beamlines built at NSLS-II from the beginning. We envision the development of portable and compact laser heating systems that will allow simultaneous high-pressure and temperature experiments to be performed using diamond anvil cells at nearly any beamline. Our experience at other synchrotron sources is that new X-ray techniques are commonly adaptable to high-pressure application, but this often requires restructuring of the sample stage and/or X-ray optics. However, the special needs of high-pressure equipment need to be considered now, in the design phase of the experimental stations for NSLS-II. This will be of great benefit, enable a greater array of groundbreaking scientific research, and ultimately reduced cost.

The NSLS-II x-ray sources offer great improvements over the existing NSLS beamlines for experiments at extreme conditions. These improvements arise from the following four factors:

1. The storage ring emittance is much smaller, meaning that the source size is smaller. The source divergence is also less, which is a significant factor for undulator beamlines, although not for bending magnet or wiggler beamlines, where the natural opening angle of the synchrotron radiation dominates the divergence.
2. The number of insertion device straight sections is increased by more than a factor of 4, and the maximum length of insertion devices is increased from 4.5 to 7 meters.
3. The ring current is increased from 300 to 500 mA.
4. The ring energy is increased from 2.8 GeV to 3.0 GeV.

There are three figures of merit that are commonly used when comparing synchrotron sources:

1. Flux: This is the number of photons per 0.1% energy bandwidth, integrated over the full vertical opening angle, per 1 mrad of horizontal angle. Flux is the appropriate figure of merit for a beamline with optics that can collect a large fraction of the output of a wiggler or bending magnet source. It is not very useful for high-pressure beamlines, because the high energies and small beam sizes required preclude collecting a large solid angle.
2. Intensity. This is the flux density in the center of a synchrotron beam, i.e. the number of photons per 0.1% energy bandwidth, per mrad^2 of solid angle. It is the appropriate figure of merit for a beamline with optics consisting of a small slit to define the beam, and that does not use focusing optics. An example would be white beam in the multi-anvil press. Figure 6 shows the intensity of a number of synchrotron sources, including NSLS-II undulators and wigglers, NSLS X17, APS undulator A, and the APS bending magnet.
3. Brightness. This is the intensity per unit source size. It is the appropriate figure of merit for a beamline that has focusing optics. Figure 7 shows the brightness of the same synchrotron sources plotted in Figure 6. Note that because NSLS-II has

a very small source size, it has a very large brightness. In order to take deliver this brightness to the experiment, it will be necessary to have x-ray optics of extremely high quality.

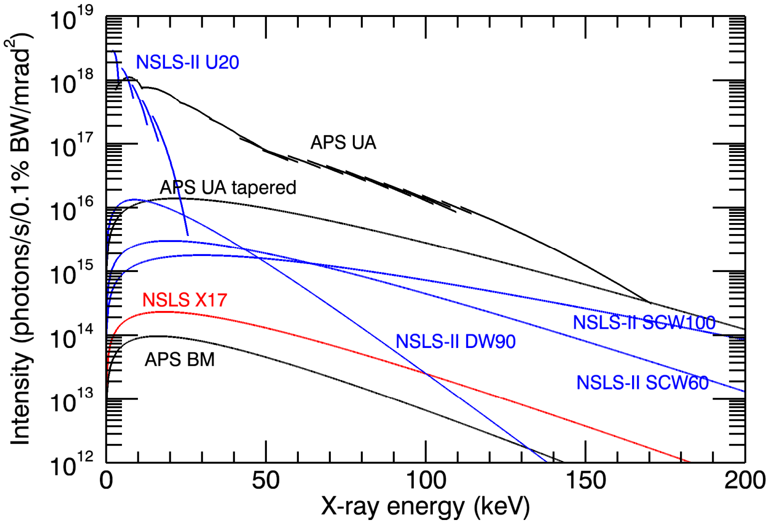

Figure 6 Intensity of synchrotron x-ray sources. These include NSLS-II 20 mm period undulator tuning curve; NSLS-II superconducting wiggler 100 mm period, 6T field; NSLS II superconducting wiggler 60mm period, 4T field; NSLS-II damping wiggler, 90 mm period; NSLS X17 superconducting wiggler; APS 33 mm period undulator tuning curve; same APS undulator with gap tapered from 10.5 to 12.5 mm, modeled as a wiggler source; APS bending magnet.

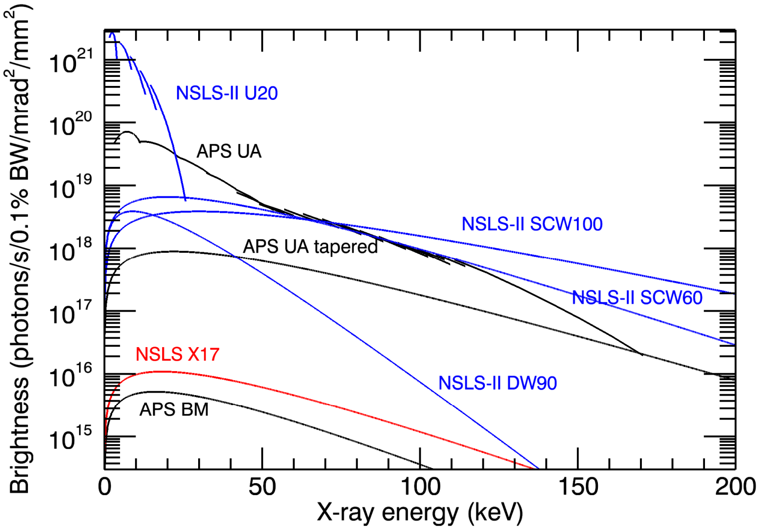

Figure 7 : Brightness of the same synchrotron x-ray sources plotted in Figure 6.

Table 1. Parameters of synchrotron sources plotted in Figures 1 and 2

Source	E (GeV)	Current (mA)	Magnetic field (T)	Period (mm)	# poles	Source size (σ_x, σ_y, μm)
NSLS-II undulator (U20)	3.0	500		20	300	28, 2.6
NSLS-II damping wiggler (DW90)	3.0	500	1.8	90	150	99, 5.5
NSLS-II superconducting wiggler (SCW100)	3.0	500	6.0	100	20	28, 2.6
NSLS-II superconducting wiggler (SCW60)	3.0	500	4.0	60	34	28, 2.6
NSLS X-17 superconducting wiggler	2.8	300	4.2	174	5	307, 11
APS bending magnet	7.0	100	6		1	109, 27
APS undulator A	7.0	100		33	144	275, 9
APS undulator A (tapered)	7.0	100	0.81	33	144	275, 9

Multi-anvil press experiments:

There are two superconducting wigglers that could be considered for the multi-anvil press high pressure beamline at NSLS-II. The first is a 6 T device with a 100 mm period, and the second is a 4 T device with a 60 mm period. The lower field device can have more periods (at a fixed 1 m length), and thus higher intensity up to about 65 keV. The lower field device also has many fewer photons at very high energy (e.g. 300 keV), which could greatly simply the shielding in the hutch when using white beam. These wigglers provide an intensity that is 10-20 times greater than X17 up to 100 keV. Given that the experiments at NSLS-II will be about twice the distance from the source as those at NSLS, the gain in photons on the sample through a slit will be about 2.5 to 5 over the existing setup at X17B2. The fact that the source size is much smaller at NSLS-II will not have a significant impact on these experiments, since they do not use focusing optics. Relative to the APS undulator operated in tapered mode, the NSLS-II will be a factor of 2 to 10 lower in intensity, depending on the energy and which wiggler is selected.

Diamond anvil cell experiments:

The diamond cell experiments at NSLS-II will use focusing optics, and so brightness is the appropriate figure of merit (Figure 7). It can be seen that NSLS-II superconducting wigglers offer increases of more than 300 relative to X17. Even more impressive are the gains from the U20 undulator at NSLS-II. This has a brightness up to 5 orders of magnitude greater than X17, and the brightness exceeds that of the NSLS-II superconducting wigglers up to about 30 keV. Indeed the U20 undulator has a higher brightness than the APS undulator up to about 25 keV. The U20 undulator will be an excellent source for spectroscopy, inelastic scattering, and diffraction below about 25 keV. It will offer a tremendous increase in capabilities relative to X17. The large gap (90 mm) bending magnet is the ideal source for the proposed far-infrared beamlines. It will provide better flux and brightness over the entire IR region and a 10-1000 times better stability. This will enhance the experimental capabilities for IR spectroscopy at extreme conditions.

A major challenge will be the development of optics that can take advantage of the very small source sizes at NSLS-II. For example, in order to preserve the brightness of the source an optic such as a Kirkpatrick-Baez mirror must have slope errors that are less than about 25% of the angular size of the source as viewed from the optic. At NSLS-II the vertical source size for the undulator is 2.6 microns, and the optic will be about 50 m from the source. The slope error requirement is thus $0.25\times2.6\times10^{-6}/50 = 1.3\times10^{-8}$. This is a slope error of 0.013 μrad. The best mirrors we are currently able to purchase have slope errors of about 0.50 μrad, so factors of 40 improvements in quality are required.

In summary, the above discussion illustrates that the three proposed extreme conditions beamlines for extreme conditions will provide unique capabilities currently not available in the portfolio of DOE beamlines at synchrotron radiation facilities.

Superconducting Wiggler

Diamond Anvil Cell Stations

The high pressure community identified the need for two extreme conditions diffraction stations utilizing small pressure generating devices like diamond anvil cells (DAC) or small Paris-Edinburgh (PE) cells, located at the superconducting wiggler port. The experimental capabilities of the two diffraction beamlines are complementary to each other and to other diffraction beamlines proposed for NSLS-II.

Station A

Station A will be a diffraction beamline for experiments at simultaneous high pressure and temperature. An experimental hutch of sufficient size (4×5 m) to accommodate the experimental setup including a permanent laser heating system and possibly other spectroscopic (Raman, Brillouin) systems is necessary. The station will operate at a fixed energy in the range of 35-40 keV and will be optimized for the following high-demand experimental techniques:

- **Powder diffraction:** Measurements of PVT equations of state, compressibility, structural evolution, phase transformation, element partitioning, melting, strength, rheology, etc of simple to moderately complex structures at pressure and temperature. Time resolved measurements of phase transformations and reactions to determine kinetic parameters.

- **Single crystal diffraction:** Determination of complex crystal structures and the investigation of their compression behavior.

- **Quasi single-crystal diffraction:** The micro-beam capabilities will make a new class of experiments at high pressure and temperature possible. Experiments at high pressure and temperature lead often to transitions of a single phased compound to a multi phase assemblage. The micro-beam will allow to probe desired grains in the polycrystalline and multiphase sample and perform single crystal diffraction on these grains with sizes $< 1\mu m$.

The following components for the beamline and the experimental station are proposed:

- The monochromator will be a side scattering asymmetric bent Laue crystal utilizing the (111) or (311) reflection of silicon. To gain a high stability the monochromator needs to be liquid N_2 cooled. The energy resolution of the monochromator should be aimed for $\Delta E/E$ of 1×10^{-3} to 1×10^{-4}.

- Microfocusing optic, including silicon kinoform Fresnel lenses, Kirkpatrick-Baez mirrors, or zone plates will be used to focus the beam in the horizontal and vertical directions. The beam size for Station A should be adjustable between 5µm and 100 nm.

- The sample stages need degrees of freedom in x, y, z, ω, φ in order to perform the above-mentioned experiments. The mechanical stability of the optical table and the translation and rotation stages needs to be high, due to the small beam size in combination with the laser heating setup. Furthermore, the sphere of confusion of the translation and rotation stages needs to very small, to meet the extraordinary demands of micro diffraction.

- An area detector will be used to detect the diffraction patterns. The use of the germanium pixel-array detector, currently under developed by D.P. Siddons at NSLS, is anticipated to be one of the detector options for Station A.

- A double-sided laser heating system suited for CO_2 and Yb:fiber laser will add high temperature capabilities to the beamline. Extrapolating the current advancements in laser technology, a very compact, but mechanically stable, design for the laser heating system might be possible.

- A micro-Raman spectrometer for simultaneous X-ray diffraction and Raman spectroscopy studies will be installed at the beamline. This system will be used for online pressure measurements by fluorescence methods as well.

- Additional the following beamline components will be needed: Slit systems, cleanup slits and pinholes, motorized beam-stop, cameras, and ionization chambers.

Station B

Station B will be a diffraction beamline for experiments at simultaneous high pressure and high/low temperatures. An experimental hutch of sufficient size (3×5 m) to accommodate the experimental setup including the laser heating systems and a cryostat is necessary. The station will operate in the energy range from 20-120 keV. The wide energy range will allow large variety of scattering experiments at extreme conditions, currently not possible at other dedicated high-pressure beamlines in the world. In addition to conventional powder and single crystal diffraction at extreme conditions, Station B will allow the following experimental techniques.

- **X-ray total scattering:** Standard X-ray diffraction techniques that are only take the Bragg component of the elastic scattering into account for structure determination fail on whole classes of materials (e.g. nanocrystalline, amorphous materials and liquids) because of their limited structural coherence. Total elastic X-ray scattering, including the Bragg and diffuse contributions, in conjunction with pair distribution function analysis allows determining the short-, intermediate- and long-range atomic arrangement. A high-energy incident X-ray beam with large detector coverage is necessary to measure high wave vector transfers in order to obtain a real space resolution of better than 1 Å.

- **Resonant scattering:** The high brilliance of NSLS-II will allow measurements of the anomalous dispersion of the structure factor close to the *K*- and *L*- absorption edges at extreme conditions. Gaining additional information of the crystal structure of complex crystalline and non-crystalline materials at extreme conditions. The energy range of the proposed experimental station will allow resonant scattering experiments on absorption edges of elements with $Z > 43$.

The following components for the beamline and the experimental station are proposed:

- The monochromator needs to cover a wide energy range and the energy needs to be tunable. Two monochromators are needed to fulfill these requirements. A silicon (111) double crystal monochromator (Bragg geometry) for the energy range of 20-50 keV and a silicon (311) sagitally bent Laue double crystal monochromator for the energy range above 50 keV.

- Mirrors in Kirkpatrick-Baez geometry will achieve horizontal and vertical focusing. The beam size for Station B should be between 1-5 μm.

- The sample stages need degrees of freedom in x, y, z, ω, χ, φ, 2θ in order to perform the above-mentioned experiments. The mechanical stability of the optical table and the translation and rotation stages needs to be high, due to the small beam size in combination with the laser heating or cryostat setup.

- An area detector will be used to detect the diffraction patterns. The use of the germanium pixel-array detector, currently under developed by D.P. Siddons at NSLS, is anticipated to be one of the detector options for Station A. Single crystal diffraction measurements and resonant scattering experiments will benefit from the availability of a point detector, usable with and without analyzer crystal.

- A double-sided laser heating system suited for CO_2 and Yb:fiber laser will add high temperature capabilities to the beamline. Extrapolating the current advancements in laser technology, a very compact, but mechanically stable, design for the laser heating system might be possible.

- A closed cycle He-cryostat capable of reaching temperatures of < 1 K will add low temperature capabilities to the beamline.

- A micro-Raman spectrometer for simultaneous X-ray diffraction and Raman spectroscopy studies will be installed at the beamline. This system will be used for online pressure measurements by fluorescence methods as well.

- Additional the following beamline components will be needed: Slit systems, cleanup slits and pinholes, motorized beam-stop, small crane for moving heavy equipment (e.g. cryostat), cameras, and ionization chambers.

Large Volume Press Stations

The high pressure community identified the need for two extreme conditions diffraction stations with large hydraulic presses to generate sample environments at extreme pressure and temperature in situations where large samples (1 mm) are required or uniform pressure and temperature are important. These systems will generally work at pressures up to about 60 GPa.

Station C

Station C will be a diffraction beamline for experiments at simultaneous high pressure and temperature. An experimental hutch of sufficient size (4×5 m) to accommodate the experimental setup and space for several large Paris-Edinburgh type pressure cells is necessary. The station will operate at a fixed energy in the range of 35-40 keV and will be used to explore samples at extreme conditions using X-ray diffraction and imaging. Interchangeable high pressure systems will be used here. The different systems will be optimized for a variety of experimental goals.

- **Slow dynamic processes:** Materials respond to pressure, temperature and stress on various time scales. Several time-resolved experiments require long periods of time to define kinetics or rheology at slow strain rates. This hutch will allow a high pressure experiment to continue over several days/weeks but not continually be in the X-ray beam. The experiment can be started, be characterized with X-rays and then continue at high pressure and temperature off-line while other experiments take the beam time. In certain time intervals, the pressure cell will be moved onto the experiment and characterized with X-ray techniques.

- **Sample imaging:** Cells with a large angular access will be used for tomographic imaging of the sample. This will allow studies of fluid flow through the sample at elevated pressure and temperature. Melting phenomena can also be studied with this technique.

- **Powder diffraction:** Equation of state measurements, phase transformations, kinetics can be studied with diffraction measurements.

The following components for the beamline and the experimental station are proposed:

- The monochromator will be a side scattering sagittal-focusing asymmetric bent Laue crystal utilizing the (111) or (311) reflection of silicon. To gain a high stability the monochromator needs to be liquid N_2 cooled. The energy resolution of the monochromator should be aimed for $\Delta E/E$ of 1×10^{-4}, since we expect a major advancement in the spatial resolution of area detectors. The focusing capacity of the monochromator will be sufficient for a beam size of about 100 μm.

- Detector: an area detector will be primarily used in this hutch. Also available will be a high-resolution detector system with two detectors, one about a two theta axis that is vertical, the other horizontal. This will enable high accuracy stress measurements.

- Paris-Edinburgh type high pressure cells with variable pressure modules designs. Opposed Anvil, T-Cup, DT-Cup. Clamp cells with cryostat.

- Additionally, the following beamline components will be needed: Slit systems, cleanup slits and pinholes, motorized beam-stop, small crane for moving heavy equipment (e.g. PE cell), cameras, and ionization chambers.

Station D

Station D will house a 2000-ton hydraulic press with interchangeable high pressure toolings. These toolings will be specialized to serve several different experiments where a high pressure – high temperature environment is important. This provides a versatile experimental environment that can continually expand as new needs arise by the design and implementation of new tooling sets. The station will operate in the energy range from 20-120 keV. Both monochromatic and white x-rays will be available for the experiments as some high pressure configurations have very limited angular access for the detection. The wide energy range will allow large variety of scattering experiments at extreme conditions, currently not possible at other dedicated high pressure beamlines in the world.

- **Rheology:** The strength of material at high pressure and temperature – and the viscosity of solids can be studied using deformation tooling. A combination of diffraction and imaging provide the needed data for this information.

- **Ultrasonic elastic properties:** Elastic properties are fundamental properties of materials that map into the equation of state and further define the response to stress. Acoustic waves sample this information, but require mm sized samples.

- **Tomographic imaging:** Phenomena from fluid flow to faulting are possible to study with 3-d mapping of the sample with time. By doping the sample with materials that have x-ray contrast allows these techniques to be applied at high pressure and temperature.

Beamline with monochromatic and white beam capabilities

- 2000 t hydraulic jack and frame
  - Kawai style system: routine capable of 30 GPa and 3000K, but may reach 60 GPa and 3000K, for 1×1×1 mm^3 dimension samples.
  - DDIA Deformation system. Capable of 10 GPA and 3000K with uniaxial stress capability, for 1×1×1 mm^3 samples
  - Deformation Kawai style, pressure of Kawai device with uniaxial stress capability. Currently under development.
  - Rock mechanics imaging system: 1 GPa pressure, 1000K 10x10x10 cm samples for fluid flow studies using tomographic imaging.
  - Rotational Drickamer device: 50 GPa, 3000K with shearing stress, 3×1×1 mm^3 sample. Also useful for tomographic imaging at high pressure.
- The monochromator needs to cover a wide energy range and the energy needs to be tunable. Two monochromators are needed to fulfill these requirements. A silicon (111) double crystal monochromator for the energy range of 20-50 keV and a silicon (311) sagitally bent Laue double crystal monochromator for the energy range above 50 keV.
- Focusing should be available for both white and monochromatic beams. Spot sizes from 0.5 mm to 0.005 mm are required. The small size will be used to investigate lateral variations within the large sample.
- Imaging requires a parallel incident beam and an expansion of the transmitted beam. The optics for this need to be developed.
- Diffraction. High d-spacing resolution in multiple azimuthal directions is required for stress measurements. Goals of 10^{-6} are required for d-spacing resolution.

Undulator U20

The rapid development of inelastic scattering techniques provides a multitude of probes of elementary excitations in condensed matter in a broad energy and momentum parameter space. High resolution techniques usually require tight focusing of X-rays, which is ideally matched to the experimental conditions in DAC to very high pressures in excess of 300 GPa The experimental station proposed here is specialized on a suite of techniques, which matches ideally the future NSLS-II coherent sub-meV resolution x-ray sources. The brightness of the X-rays at NSLS-II is an order of magnitude greater than at the APS (few keV to 20 keV range), are ideally matched to the multitude of high pressure spectroscopic techniques thriving at 3rd generation synchrotron sources.

The high pressure community proposes one experimental station at the undulator U20 port which will be specialized on X-ray spectroscopy at extreme conditions. Although the energy range provided by the undulator is not ideally suited for diffraction experiments at extreme conditions, some diffraction capabilities should be available at this beamlines to allow characterization of the same sample by spectroscopic and diffraction methods.

Station A

Station A specializes on X-ray spectroscopy experiments at simultaneous high pressure and high/low temperatures. An experimental hutch of sufficient size (4×6 m) to accommodate a large goniometer and equipment to generate high and low temperatures is necessary. The station will operate in the energy range from 5-25 keV and will be optimized for the following techniques.

- **X-ray absorption spectroscopy:** X-ray absorption spectroscopy can be divided into near edge spectroscopy (XANES) and extended X-ray absorption fine structure (EXAFS). In the EXAFS region, the excited electron has significant kinetic energy and EXAFS spectrum contains information on the local geometry around the absorbing atom. The XANES structure can be described be the multiple scattering, or alternatively, one can use electronic structure models such as density-functional theory to calculate the unoccupied density of states (DOS). X-ray magnetic circular dichroism (XMCD) is an important phenomenon in both X-ray absorption and X-ray emission. The magnetic structure of a system is studied by making use of circular polarized X-rays. XMCD can be observed in both XANES and EXAFS. XMCD in XANES can measure spin-resolved conduction band densities of states, whereas XMCD in EXAFS provides local magnetic structural information. The unique advantage of NSLS-II for the suite of high-pressure EXAFS, XANES, and XMCD is in tightly focused x-ray beams, which will allow detailed analysis of the samples in the high-pressure environment by mapping of local structure, valence states, magnetic structure, and spin-dependent density of states with high spatial resolution at ultra-high pressures.

- **X-ray near-edge spectroscopy:** Near core-electron absorption edge features measured by soft x-ray absorption (XANES) or electron energy loss spectroscopy (EELS) reveal information on chemical bonding. Such information is particularly pronounced and important for light elements, but has been inaccessible for high-pressure studies as the pressure vessel completely blocks the soft x-ray and electron beams. With x-ray inelastic near-edge spectroscopy (XINES), the high-energy incident x-ray penetrates the pressure vessel and reaches the sample. The scattered photon loses a portion of energy corresponding to the *K*-edge of the low-Z sample, but can still exit the vessel to be registered on the analyzer-detector system. Inelastic *K*-edge scattering spectra of second-row elements from Li (56 eV) to O (543 eV) at high pressures opened a wide new field of near *K*-edge spectroscopy of the second row elements.

- **X-ray emission spectroscopy:** In the x-ray emission (XES) technique, deep-core electrons in the sample are excited by x-rays. The core-holes then decay through either radiative or non-radiative processes. For deep-core holes, the dominant decay channels are radiative processes, producing fluorescence, which is analyzed to provide information on the filled electronic states of the sample. The information provided by XES is complementary to that provided by x-ray absorption spectroscopy. The final state of the fluorescent process is a one-hole state, similar to the final state of a photoemission process. Thus, the important information provided by photoelectron spectroscopy, namely large chemical shifts in the core-level binding energies and the valence band density of states, is available in XES.

- **Nuclear resonant inelastic X-ray scattering:** The inelastic method provides specific information about materials vibrational states, e.g., the phonon density of states. The Mössbauer method is a technique of choice to measure hyperfine interactions. All nuclear resonance techniques take full advantage of the unique properties of synchrotron radiation: intensity, collimation, time structure, and polarization. As a result both methods have led to novel applications for materials under extreme conditions. Nuclear resonant scattering yields information on the phonon density of states (DOS) through an inelastic scattering. In principle, the DOS provides constraints on dynamic, thermodynamic, and elastic information of a material, including vibrational kinetic energy, zero-point vibrational energy, vibrational entropy, vibrational heat capacity, Debye temperature, Grüneisen parameter, thermal expansivity, longitudinal velocity, shear velocities, bulk modulus, and shear modulus.

- **Nuclear forward scattering:** Mössbauer spectroscopy has been used extensively in high-pressure mineralogy in laboratory studies with a radioactive parent source. High-pressure studies using a conventional Mössbauer source suffer from limited intensity for measurements on small samples, absorption by anvils, and background scattering. The nuclear forward scattering can be used to measure magnetic transitions and hyperfine fields at high and low temperatures, and to probe valence changes under pressure and temperature.

The following components for the beamline and the experimental station are proposed:

- A cryogenically cooled monolithic silicon (111) monochromator will be used at the beamline, giving an energy resolution of 1eV. An additional cryogenically cooled double channel-cut silicon monochromator will be employed for experiments, which need a higher energy resolution (2meV).

- Large mirrors in Kirkpatrick-Baez geometry will allow to collect nearly the entire fan emitted by the undulator. The beam can be focused to a size of about 10 µm with this set of mirrors. For experiments that need a smaller focal size, a set of small mirrors in Kirkpatrick-Baez geometry will be available, allowing a beam size of 1 µm.

- A variety of detectors and analyzers should be available for the different inelastic X-ray scattering techniques depending on the needed energy resolution. We anticipate the need for a silicon pixel-array detector, a multi-element silicon drift diode for fluorescence measurements and a multi-crystal analyzer. Furthermore, a CCD or germanium pixel-array will serve as detector for diffraction experiments.

- A large 6-circle Kappa-geometry diffractometer, which can accommodate heavy equipment on the detector arm (e.g. detector and multi-crystal analyzer) and on the sample position (e.g. cryostat), will be installed.

- A double-sided laser heating system suited for Yb:fiber laser will add high temperature capabilities to the beamline. Extrapolating the current advancements in laser technology, a very compact, but mechanically stable, design for the laser heating system might be possible.

- A closed cycle He-cryostat capable of reaching temperatures of < 1 K will add low temperature capabilities to the beamline.

- Additional the following beamline components will be needed: Slit systems, cleanup slits and pinholes, motorized beam-stop, small crane for moving heavy equipment (e.g. cryostat), cameras, and ionization chambers.

Bending Magnet

The beamline for infrared spectroscopy at extreme conditions should be located on a bending magnet port with a wide gap (90 mm) dipole. The energy range should cover the far infrared to visible spectrum. An experimental hutch of 4×6 m is needed to accommodate the experimental setup. The interlock system should allow for simultaneous operation of IR spectroscopy station, an ion Argon and a high power CO_2 lasers for the laser heating system. The hutch should be located nearby the high-pressure undulator X-ray beamlines to create the opportunity to connect the IR beam through an extension pipe into the undulator hutches in order to perform *in si*tu X-ray and IR studies for same samples under extreme conditions. Beside standard IR spectroscopy at extreme conditions, a large growth potential lies in the following two experiments that will be facilitated by the unique beam characteristic of NSLS-II.

- **IR spectroscopy at simultaneous high pressure and high temperature:** Currently, IR spectroscopy measurements at high pressure are limited to temperatures of 1000K or investigations of samples quenched after offline laser heating. The high flux and the high spatial resolution of NSLS-II will allow performing IR spectroscopy measurement on samples heated simultaneously by a CO_2 laser. This unique capability will open exiting new research directions in Earth and material sciences.

- **IR spectroscopy coupled with dynamic compression:** Material emissivity measurements at extreme conditions can provide fundamentally important data that allow the measurement of temperature on short time scales, which is crucial for the complete characterization of dynamic compression events. For opaque materials such as metals, reflectivity measurements are necessary and must be conducted under dynamic compression in order to provide the necessary information. Although synchrotron radiation has been extensively adapted to different static high-pressure techniques for many years, the time-resolved capability from a pulsed synchrotron source has never been utilized to study the optical properties for materials under dynamic compression.

It is anticipated that the current IR spectroscopy end station U2A will be further upgraded and finally be moved to NSLS-II after the shutdown of NSLS. The move of the complete infrared end station can be accomplished in 4-6 weeks; therefore, the high pressure IR beamline will serve the extreme conditions community at NSLS until the last photons are emitted.

Ancillary Laboratories and Office Space

The support laboratories are almost as important as state of the art synchrotron radiation beamlines for a successful high pressure program at NSLS and NSLS-II. The sample and cell preparation for diamond anvil cell and large volume press experiments is space intensive and needs various specialized equipment. The high pressure laboratory will not only be supporting the dedicated high pressure beamlines, but function as a home for the high pressure research around the NSLS-II ring. Beside the laboratory facilities, lined out in detail below, sufficient office space for the beamline personnel is needed. Furthermore, since the current ring design is lacking space for beamline control and data collection areas at the beamlines, beamline control rooms are needed in the Laboratory and Office Building.

- Sample preparation laboratory (50 m^2)
    - 4 Stereo zoom microscopes with cameras and monitors
    - Workbench with granite surface
    - Fume hood
    - Glove box
    - 3 Furnaces (regular and vacuum)
    - Refrigerator
    - Microwave
    - 2 Ultrasonic baths
    - Small hydraulic press
    - Buffing & polishing machine
    - Diamond saw and cutting equipment
    - Balance
    - Heating plates
    - Micro drilling machines (spark erosion, mechanical, laser)

- Pressure medium loading laboratory (25 m^2)
    - Cryogenic loading equipment
    - Gas loading apparatus

- Laser laboratory (25 m^2)
    - Raman spectrometer (Ruby fluorescence and standard Raman measurements)
    - Offline laser heating station (Yb:fiber laser, CO_2 laser)

- User machine shop (25m^2)
    - Lathe
    - Polishing and buffing machine
    - Drilling machine
    - Thermocouple welder

- Staging and storage area (50m^2)

In the next decade, micro-engineering of the diamond anvil cell sample environment promises to undergo major development. Also, advanced methods for chemical analysis of recovered samples also offer great promise that complement structural probes. These are areas with potential synergistic interactions with the Center for Functional Nanomaterials (CFN) at Brookhaven National Laboratory. We propose that a Diamond anvil cell micro-preparation and nano-analysis facility be established at CFN and NSLS-II. This may include such capabilities as CVD growth of designer anvils, ion implantation, micro- and nano-scale fabrication of sample assemblages, as well as material synthesis and characterization capability. For chemical analysis of recovered samples, focused ion beam milling systems combined with TEM or nano-SIMS devices will be increasingly required in the coming decade for a complete characterization of the chemical and structural states of materials achieved under extreme conditions.

High Pressure Working Group

During the scientific planning workshops for NSLS and NSLS-II and the technique-based workshops, many scientific communities expressed interest in high pressure sample environments. High pressure research was a topic in many of the technique based workshops and the desire to incorporate high pressure capabilities in many of the six project beamlines was shown. A large variety of high pressure cells are portable and can be installed at beamlines not dedicated to high pressure. However, certain choices in the design of a beamline, which would prohibit the use of high pressure sample environments, need to be avoided. Ideally, a representative of the high pressure community would be a member on the Beamline Advisory Team (BAT), for each beamline interested in allowing high pressure research. However, currently just the Inelastic X-ray Scattering Beamline and the Powder Instrument New Generation have members with a background in high pressure research on their BAT. Therefore, the high pressure community formed the “High Pressure Working Group” for NSLS-II.

The two main functions for the members of the working group are:

1. The “High Pressure Working Group” will serve as point of contact for BATs, providing in depth knowledge of high pressure instrumentation. The members can advice on how to best integrate high pressure equipment in beamline designs in order to optimize the research capability

2. Creating a synergy effect with other BNL institutions. Several BNL institutions (e.g. CFN, CMPMSD) possess experimental capabilities and instruments that would be useful for a sample characterization after a high pressure experiment. The “High Pressure Working Group” can initiate contact with these institutions and develop a plan to integrate these experimental capabilities in the high pressure program at NSLS-II.

The “High Pressure Working Group” is comprised of the following members:

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