1st Workshop for the Extreme Conditions Beamline at PETRA III

18-19 May 2009

Notkestraße 85, 22607 Hamburg, Building 28c, FLASH Auditorium

Arial view of the new PETRA III experimental hall.

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Sunday May 17th		
18:00	Pre-registration on Sunday evening at Novotel	
Monday May 18th		
8:30 – 8:45	Welcome	**H. Franz**
Session 1: Extreme Condition Research: State of the Art *Chair: H. Franz*		
8:45 – 9:30	Overview in Solid State Chemistry – The laser-heated DAC as a reaction chamber	**B. Winkler**
9:30 – 10:15	Extreme Condition Research: State of the Art in Geophysics	**D. Andrault**
10:15 – 10:30	Coffee Break	
Session 2: Existing extreme condition synchrotron facilities *Chair: H.-P. Liermann*		
10:30 – 11:15	The high pressure diffraction beamline ID09A at the ESRF	**M. Hanfland**
11:15 – 12:00	ID27, a state-of-the-art XRD beamline for research at extreme pressures and temperatures	**M. Mezouar**
12:00 – 13:15	Lunch	
13:15 – 14:00	High Pressure Research at GSECARS	**M. Rivers**
14:00 – 14:45	Advancing HP-SR Research at HPCAT and HPSynC	**Ho-kwang Mao**
Session 3: Research Focus and design of the Extreme Conditions Beamline *Chair. W. Morgenroth*		
14:45 – 15:15	Overview PETRA III	**H. Franz**
15:15 – 15:45	The Extreme Conditions Beamline at PETRA III, DESY: Possibilities to conduct time resolved monochromatic and pink beam diffraction experiments in laser heated DAC	**H.-P. Liermann**
15:45 – 16:00	Discussion	
16:00 – 16:15	Coffee Break	
Session 4: Technical Challenges for an Extreme Conditions Beamline *Chair: M. Rivers*		
16:15 – 17:00	White-beam micro Laue-diffraction with an undulator	**G. Ice**
17:00 – 17:45	Submicron diffraction at ALS beamline 12.3.2	**M. Kunz**
Session 5: Examples for Powder and Single Crystal Diffraction at simultaneous high-pressure and high/low-temperatures *Chair: NN*		

17:45 – 18:30	Tayloring electronic properties by high pressure	**U. Schwarz**
18:30 – 19:00	Poster Session & Initial Meeting of the Technical Advisory Committee	
19:00	Workshop Dinner	
Tuesday May 19th		
8:30 – 8:45	Announcements and Miscellaneous	**H.-P. Liermann**
Session 6: Dynamic Single Crystal Diffraction at simultaneous high-pressure and -temperatures *Chair: B. Winkler*		
8:45 – 9:30	Single crystal diffraction at ultrahigh pressure	**P. Dera**
9:30 – 10:15	Laser-heating and synchrotron measurements	**R. Boehler**
10:15 – 10:30	Coffee Break	
10:30 – 11:15	X-ray diffraction experiments with internally and externally electrically heated DACs	**L. Dubrovinsky**
11:15 – 12:00	Structural, magnetic and electronic transitions in 3d-metal oxides under super high pressure	**I. S. Lyubutin**
12:00 – 13:15	Lunch	
Session 5 (continued): Examples for Powder and Single Crystal Diffraction at simultaneous high-pressure and high/low-temperatures *Chair: NN*		
13:15 – 14:00	Single crystal studies of elemental systems	**M. McMahon**
14:00 – 14:45	The Dynamic Diamond Anvil Cell (dDAC): A Novel Device for Studying the Dynamic Properties of Materials at High Pressure	**W. J. Evans**
14:45 – 15:00	Coffee Break	
15:00 – 15:45	Brillouin scattering at high-pressure and -temperature	**S. Speziale**
15:45 – 16:30	Plastic properties of minerals at high-pressure and -temperatures	**S. Merkel**
16:30 – 16:45	Coffee Break	
16:45 – 18:00	Discussion	**H.-P. Liermann, B. Winkler**
18:00 – 19:00	Technical Advisory Committee Meeting	

Overview in Solid State Chemistry - The laser-heated DAC as a reaction chamber

WINKLER, Björn

Goethe-Universität Frankfurt

One envisaged class of experiments at the "extreme conditions" station at PETRA III are synthesis experiments at very high pressures and temperatures. I will summarize recent work on the synthesis of carbides and nitrides in laser heated diamond anvil cells and discuss future prospects in this research area.

Extreme Condition Research: State of the Art in Geophysics

ANDRAULT, Denis

Université Blaise Pascal

Mineral Physics is a dynamical field of research, but in many aspects our progression is limited by technology. Any improvement in experimental techniques, to which we participate actively, results in opening new windows to the Earth interior. We employ a great diversity of difficult experimental techniques; the 50µm diameter samples loaded between two beveled diamond anvils, advanced optical systems for laser heating, the hundreds of tones available in large volume presses, the many devices available on a synchrotron beamlines, for examples. In fact, there is an equilibrium between being a developer of scientific methods or an opportunist user that one should find, in our field, in order to obtain the most original results. For this reason, the most efficient research tools should be adaptable and evolutive.

In the past few years, rapid evolution of synchrotron beamlines and of all devices associated to it offered great opportunities to refine our knowledge on the Earth's interior. Various type of experiments can now be performed at higher pressures, higher temperatures, for sample compositions fully relevant to the Earth, in specific configurations with mixed liquid and solid phases, under controlled stress, etc: We can measure PVT equation of state up to more than 150 GPa and 4000 K, refine crystalline structure of new phases of a few µm3, probe the phase relations and determine phase diagrams in situ, etc.

Limitations remain, however, because our samples are very small, complex, and, on the other hand, we are greatly interested in resolving better the sample properties. Therefore, our field of research call for smaller probes, higher fluxes, better spectral resolution, etc. In this talk, I will present recent results that brought new views on the deep Earth's interior and that illustrate how important are the forthcoming improvements.

The high pressure diffraction beamline ID09A at the ESRF

HANFLAND, Michael

ESRF

ID09A uses monochromatic diffraction with large area detectors on single crystals and powdered samples at high pressures in diamond anvil cells.

It provides variable beam sizes down to 10 x 10 micron, to study samples from a few GPa to approximately 200 GPa at ~30 keV and very high photon fluxes of ~10^13/sec. It offers state of the art optical systems for additional in situ characterisation of the samples at high pressure (Raman, etc.) and a highly developed sample environment.

Recently the MAR345 online image plate reader was replaced by a MAR555 flat panel detector. Advantages of the new detector will be discussed, examples of the use of the detector in powder and single crystal diffraction will be given.

ID27, a state-of-the-art XRD beamline for research at extreme pressures and temperatures

MEZOUAR, Mohamed
ESRF

This presentation is devoted to the description of a state-of-the-art synchrotron beamline fully optimised for monochromatic X-ray diffraction at high pressure and high (or low) temperature. This beamline exhibits outstanding performance in terms of photon flux and focusing capabilities at high X-ray energies. The main components of this instrument will be described in some details. In particular, the choices in terms of X-ray source, X-ray optics, detectors and sample environment will be discussed.

High Pressure Research at GSECARS

RIVERS, Mark
University of Chicago
PRAKAPENKA, Vitali
University of Chicago
DERA, Przemek
University of Chicago
WANG, Yanbin
University of Chicago

GSECARS, sector 13 at the Advanced Photon Source, operates as a national user facility for earth, planetary, and environmental research. It includes facilities for both diamond anvil cell and large volume high-pressure research. The high-pressure facilities include:

- 13-ID-D station with online laser heating on an undulator beamline. The laser system includes double-sided heating with two 100 watt Yb:fiber lasers, and single-sided heating with a 200 watt CO_2 laser. Experiments include x-ray diffraction, scattering and emission spectroscopy.
- 13-ID-D station with 1000-ton large-volume press. This apparatus includes a T-25 teacup module, and is used for diffraction, scattering and imaging experiments. It will soon include a D-DIA30 module for higher pressures and deformation experiments.
- 13-BM-D station diamond-anvil cell on the bending magnet beamline. This station has online Brillouin spectroscopy and external heating.
- 13-BM-D station with 250-ton large volume press. This press includes DIA, T-cup, D-DIA and rotational Drickamer modules. The Drickamer module is used for high-pressure computed tomography.
- Gas-loading system for loading inert gases at up to 2000 bar in the diamond anvil cell. This system provides online optical access with ruby fluorescence, for very high success rate in gas loading.
- Offline Raman, ruby fluorescence and Yb:fiber laser heating. This offline laser can be used for drilling diamond cell gaskets and other micromachining tasks.

Recent technical advances and scientific results will be presented.

Advancing HP-SR Research at HPCAT and HPSynC

MAO, Ho-kwang (David)

Carnegie Institution of Washington

A plethora of high-pressure (HP) synchrotron radiation (SR) techniques has been developed and established at the HPCAT beamline of APS to explore the rich behavior of materials under pressure. These include HP x-ray emission spectroscopy which provides information on the filled electronic states of the HP samples, HP x-ray Raman spectroscopy which probes HP chemical bonding changes of the light elements, HP electronic inelastic x-ray scattering spectroscopy which accesses the high energy electronic phenomena, including electronic band structure, Fermi surface, excitons, plasmons, and their dispersions, HP resonant inelastic x-ray scattering spectroscopy which probes shallow core excitations, multiplet structures, and spin-resolved electronic structure, HP nuclear resonant x-ray spectroscopy which provides phonon densities of state and time-resolved Mössbauer information, HP x-ray diffraction which determines the fundamental structures and densities of single-crystal, polycrystalline, nanocrystalline, and non-crystalline materials, and HP radial x-ray diffraction which yields deviatoric elastic and rheological information. These tools, integrated with hydrostatic or uniaxial media, laser and resistive heating, and cryogenic cooling, have enabled investigations of structural, vibrational, electronic, and magnetic properties over an extensive P-T range.

To take full advantage of the rapid advancing SR brilliance, resolutions (in energy, size, and time), polarization, and coherence, a single beamline like HPCAT has its technical and organizational limitations. The extreme synchrotron capabilities, including the maximum energy to hundreds of keV, the maximum spatial resolution in tens of nanometer, the maximum energy resolution to sub-meV, and the maximum time resolution to follow the advances of a shock-wave front, would require specialized beamlines incompatible with the dedicated, HP, multi-purpose beamline. A new infrastructure, HPSynC, has been established to bridge the HP research and most APS specialized beamlines, thus unleashing the full power of the multidisciplinary HP science.

The Extreme Conditions Beamline at PETRA III, DESY: Possibilities to conduct time resolved monochromatic and pink beam diffraction experiments in laser heated DAC

LIERMANN, H.-P.

Deutsches Elektronen Synchrotron (DESY), Hamburg, Germany

EHNES, A.

Deutsches Elektronen Synchrotron (DESY), Hamburg, Germany

WINKLER, B.

Department of Crystallography, University of Frankfurt, Frankfurt, Germany

FRANZ, H.

Deutsches Elektronen Synchrotron (DESY), Hamburg, Germany

WECKERT, E.

Deutsches Elektronen Synchrotron (DESY), Hamburg, Germany

MORGENROTH, W.

Department of Crystallography, University of Frankfurt, Frankfurt, Germany

BERGHÄUSER, A.

Department of Mineralogy, University of Hamburg, Hamburg, Germany

Powder x-ray diffraction experiments in laser heated diamond anvil cells (DAC) have been a standard experimental technique used at all 3rd generation extreme condition synchrotron facilities over the last decades. However, the combination of single crystal diffraction at simultaneous high-pressure and –temperature using a laser heated DAC has not been realized. This is in part because single crystal diffraction pattern created by monochromatic beam can only be collected on an area detector when the sample within the DAC is rotated, resulting ultimately in the obstruction of the laser heating beam. However, rotations of the sample can be eliminated when one uses pink beam Laue diffraction.

In this paper we describe the design of the "Extreme Conditions Beamline" P02B at PETRA III, Hamburg, Germany, that will be used to conduct both monochromatic (8-80 keV) and pink beam diffraction experiments. Attention will be drawn to the pink beam capabilities of the station and the alternate use of monochromatic and pink x-ray beams and the possibility to conducted single crystal diffraction in the laser heated DAC. We will discuss the different phases of the beamline development and additional high-pressure experimental techniques that will be offered once commissioning of the beamline is completed. The possibility of conducting time resolved experiments in the dynamic DAC (1) in conjunction with fast choppers will be discussed, i.e. pump and probe experiments in the sub second regime that could shade light on the nature of transient phase stages occurring during phase transition at simultaneous high-pressures and -temperatures.

1. Evans WJ, Yoo CS, Lee GW, Cynn H., Lipp MJ, Visbeck K. (2007) Dynamic diamond anvil cell (dDAC): A novel device for studying the dynamic-pressure properties of materials. Rev. Sci. Inst., 78, 073904.

White-beam micro Laue-diffraction with an undulator

ICE, Gene

Oak Ridge National Laboratory

High-energy 3rd generation synchrotron sources offer unprecedented opportunities for the characterization of materials. Undulator sources in particular provide more than four orders of magnitude higher peak brilliance than 2nd generation sources which opens new scientific possibilities for the study of small sample volumes and weak scattering. In this talk we describe the use of undulator radiation for micro Laue diffraction on beamline 34-ID-E at the Advanced Photon Source. Both polychromatic microdiffraction tomography and related techniques are discussed and the essential instrumentation to make these experiments possible is described. The implications of the structured undulator spectra and current and future methods to best control this structure for various experiments are also discussed. Finally we speculate on specific strategies that may be possible at PETRA III for advanced characterization of small sample volumes in extreme environmental chambers and discuss undulator and instrumentation choices.

This research funded by the Division of Materials Science and Engineering, Office of Basic Energy Science, U.S. Department of Energy. Experiments in part on beamline 34-ID-E of the Advanced Photon Source which is supported by the U.S. Department of Energy Office of Basic Energy Science.

Submicron diffraction at ALS beamline 12.3.2

KUNZ, Martin

ALS / LBNL

TAMURA, Nobumichi

ALS / LBNL

CHEN, Kai

ALS / LBNL & UCLA

A new facility for submicron x-ray diffraction and fluorescence mapping has been built on beamline 12.3.2 at the Advanced Light Source (ALS) of the Lawrence Berkeley National Laboratory (LBNL). This beamline benefits from the hard x-radiation generated by a 6 Tesla superconducting bending magnet (4.37 Tesla at tangent point). The stronger magnetic field increases the available X-ray spectrum from 14 keV to 22 keV as well as the flux of a 1 µm spot from 10^7 to ~ 5 x 10^9 (at 8 keV) photons per seconds (0.1% bandwidth) when compared to the warm bending magnet source where it was originally placed on. The increase in maximum x-ray energy improves the achievable orientation and strain resolution to 0.01 deg and 5 x 10^-5, respectively. The radiation is transferred from the superbend to the experimental enclosure by a focusing toroidal mirror. Inside the lead hutch, a pair of Kirkpatrick-Baez (KB) mirrors placed in a vacuum tank re-focuses the secondary source onto the sample position. A new bending mechanism allows for more reproducible and stable mirror bending and thus mirror-focusing. Focus spots around 1 µm are routinely achieved and allow a variety of experiments which have in common the need of spatial resolution. The effective spatial resolution (~ 0.2 µm) is limited by a convolution of beam size and sample stage resolution. A four-bounce monochromator consisting of two channel-cut Si(111) crystals placed between the secondary source and KB-mirrors allows for easy changes between white-beam and monochromatic experiments while maintaining a fixed beam position. High resolution sample scans are performed while recording a fluorescence signal or an x-ray diffraction signal generated by either a monochromatic or a white focused beam. The former allows for chemical mapping and texture measurements, whereas the latter is used to produce 2-d phase-distribution-, orientation-, texture- and strain/stress-maps. Accurate sample positioning onto the x-ray focus spot is achieved with a commercial laser-triangulation unit. A Si-drift detector coupled to a multi-channel analyzer serves as a high-resolution fluorescence

detector. A CCD area detector is utilized as diffraction detector. Diffraction can be performed in reflecting or transmitting geometry. Diffraction data are processed using XMAS, an in-house written software package for Laue and monochromatic microdiffraction analysis.
Future developments include the development of experimental and data-reduction tools to interpret intensities from Laue single-crystal diffraction and the expansion from 2-d mapping to depth resolved 3-d x-ray microdiffraction.

Tayloring electronic properties by high pressure

SCHWARZ, Ulrich

MPI für Chemische Physik fester Stoffe

The combination of high pressures and high temperatures is a well-known strategy in target-oriented chemical syntheses to meet the stability fields of new compounds and, thus, to synthesize solids with novel and often unusual physical properties. Beside the potential for preparative chemistry, the state variable pressure can induce structural and electronic changes of condensed phases. Magnetic transitions and spin as well as charge ordering phenomena are associated with small energy changes which often require the simultaneous realization of low-temperatures in experimental investigations. Studies on phase transformations of selected elements and intermetallic compounds will illustrate the interplay between electronic and structural alterations as evidenced by x-ray diffraction experiments at third generation synchrotron sources.

Single crystal diffraction at ultrahigh pressure

DERA, Przemyslaw

GSECARS, University of Chicago

This talk will review the main concepts, applications and capabilities of different non-conventional varieties of single-crystal X-ray diffraction (SXD) experiment utilizing synchrotron radiation for applications in ultra-high-pressure research. Examples of how such experiments can be designed and performed to best answer the scientific goals of the study and, at the same time overcome the main technical limitations imposed by the high-pressure device and type of measurement will be discussed. The emphasis will be placed on experiments that cannot be performed using laboratory instruments, e.g. involving ultrahigh (>50 GPa) pressures, poor quality samples, laser heating in diamond anvil cell (DAC), etc. The main goal of the presentation is to demonstrate that even a non-expert crystallographer, with good understanding of the general basic principles of synchrotron SXD experiments in a DAC can successfully use these techniques as invaluable and quite easy tools in his/her own high-pressure research.

Laser-heating and synchrotron measurements

BOEHLER, Reinhard
MPI für Chemie
Aquilanti , G.
ESRF and Sincrotrone Trieste
Trapananti, A.
Dipartimento di Fisica, Università di Camerino and Sincrotrone Trieste
Mezouar, M.
ESRF

Previous melting experiments in the laser-heated diamond cell at megabar pressures relied on optical observations of motion or texture. Most of these measurements yielded melting temperatures in good agreement with low-pressure data and with shock melting points obtained at high pressures except for transition metals. In order to understand this discrepancy a systematic study of these elements using X-rays was started. For this purpose, a compact, double-sided laser-heating system for diamond-cell synchrotron applications was built. The pre-aligned optical table, containing laser, spectrometer and all optics for visual observation and measuring temperatures and pressures can be set up at most synchrotron beamlines within about one hour. We carried out measurements on iron, molybdenum and tungsten up to over one megabar and up to over 4000 K at the X-ray diffraction beamline ID 27 at the European Synchrotron Facility (ESRF) and obtained first spectra of molten iron at the X-ray absorption beamline ID 24.

X-ray diffraction experiments with internally and externally electrically heated DACs

DUBROVINSKY, Leonid

BGI

DUBROVINSKAIA, Natalia

Heidelberg University

High-pressure studies provide crucial information for many scientific and industrial applications. Volume changes can produce structural, electronic, magnetic and other phase transitions, initiate chemical reactions, and many other phenomena. During last decades diamond anvil cell (DAC) technique has become the most popular method of pressure generation capable for work in a multimegabar pressure range. However, there still are a number of problems related to high-temperature experiments with DACs. External and internal electrical heating allows generating temperature in a wide range (form 300 K to over 2500 K), relatively easy measuring it (with thermocouples at T<1500 K or spectroradiometrically at higher temperature), maintaining it practically constant (±5 K at 1500 K) during hours. Moreover, in externally heated DAC at T>800 K stresses are practically absent and heating is quite homogeneous. In other words, electrical heating could be a perfect complementary to the laser heating method to study materials at extreme conditions. We demonstrate application of electrically heated DAC on the examples of X-ray diffraction, XANES, NFS and NIS studies of geophysically important materials.

Structural, magnetic and electronic transitions in 3d-metal oxides under super high pressures

LYUBUTIN, Igor S.
Shubnikov Institute of Crystallography, Russian Academy of Sciences, 119333 Moscow, RUSSIA

Transition metal oxides constitute a very large class of materials of considerable importance for both fundamental science and practical applications. They include high temperature superconductors, manganites with colossal magneto-resistance, multiferroics, materials for spintronic and optoelectronic applications. Moreover, mixed iron oxides and perovskites are very important for geophysics. The properties and behavior of transition metal oxides are mainly governed by strong electron-electron correlations. In these systems many theories predict the pressure-induced insulator-metal phase transition which is accompanied by a collapse of the localized magnetic moment and a structural phase transition. However, until recently such scenario was not observed experimentally, since the expected values of necessary pressures exceed 2 Megabar.

We present the complex studies performed at super high pressures up to 200 GPa in diamond anvil cells using several experimental methods: Mössbauer transmission spectroscopy and Mössbauer synchrotron spectroscopy (Nuclear Resonance Forward Scattering – NFS), X-ray diffraction and synchrotron high resolution X-ray emission Fe-K β spectroscopy (XES), optical absorption spectroscopy and direct measurements of electric resistance. The materials with different crystal structures, such as nickel monoxide NiO and magnesiowüstite (Mg,Fe)O), iron oxide Fe_2O_3, rare-earth orthoferrites $RFeO_3$ (R = Nd, Lu, Y), yttrium iron garnet $Y_3Fe_5O_{12}$, iron borates $FeBO_3$ and $GdFe_3(BO_3)_4$, multiferroic $BiFeO_3$ and manganites $LaMnO_3$ and $MnFeO_4$ have been studied.

The transitions from the magnetic to a nonmagnetic state (magnetic collapse) was discovered in all crystals at pressures of about 40-55 GPa. The magnetic collapse is accompanied by a transformation of electronic and spin structures due to the spin crossover in 3d electron system at the transition of Fe^{3+} ions from the high-spin S = 5/2 (HS) to the low-spin S = 1/2 (LS) state. At the same pressures, the structural phase transitions with a sharp drop of the unit cell volume were found in several crystals. We have established that the metallization process is very complicated. In the pressure region of the HS-LS spin crossover, many crystals are transformed from the dielectric to a semiconducting state. The direct insulator-metal transition (IMT) was found in $Y_3Fe_5O_{12}$ and $BiFeO_3$ crystals. These effects are explained by the Mott-Hubbard type transitions with the extensive suppression of strong d-d electronic correlations.

A new mechanism of the insulator-metal transition in Mott-Hubbard insulators was found. This new mechanism can be initiated by high pressure and it is driven by the spin-crossover of d5 ions from HS to LS state. The spin-crossover suppresses the Coulomb correlation energy U_{eff} down to the value enabling the insulator-metal transition according to the Mott mechanism $U_{eff}/W \approx 1$ (W is the half bandwidth). Such type of Mott metallization was observed experimentally in multiferroic $BiFeO_3$. We call the new IMT mechanism as the "Hubbard energy control" mechanism, to distinguish from the well known “bandwidth control” and “band-filling” mechanisms of the IMT.

Supports by RFBR (Grants № 08-02-00897a and № 07-02-00490-a) and by the RAS Program “Strongly correlated electronic systems” are acknowledged.

Single Crystal Studies of Elemental Systems

MCMAHON, Malcolm

The University of Edinburgh

Prior to 2007, the highest pressure at which full single-crystal x-ray diffraction data had been collected and refined was 50GPa. However, building on developments at the SRS (Darsbury), we have pushed the use of such techniques first to 100GPa on Diamond [1] and, most recently, to 145 GPa at the ESRF [2]. The use of these techniques has revealed previously unimagined structural complexity in sodium [3], and the extremely high flux at the ESRF, and the micro-focussed x-ray beams, have also enabled us to collect extremely high quality data on weakly scattering samples such as Li, N_2 and O_2, including both high- and low-temperature studies. Representative results from all these studies will presented, and the prospects for future such studies on PETRA III will be discussed.

1. M.I. McMahon, E. Gregoryanz, L.F. Lundegaard, I. Loa, C. Guillaume, R.J. Nelmes, A.K. Kleppe, M. Amboage, H. Wilhelm, and A.P. Jephcoat, Proc. Nat. Acad. Sci. 104, 17297 (2007).
2. L.F. Lundegaard, E. Gregoryanz, M.I. McMahon, C. Guillaume, I. Loa, and R.J. Nelmes, Phys. Rev. B 79, 064105 (2009).
3. E. Gregoryanz, L.F. Lundegaard, M.I. McMahon, C. Guillaume, R.J. Nelmes, and M. Mezouar, Science 320, 1054 (2008).

The Dynamic Diamond Anvil Cell (dDAC): A Novel Device for Studying the Dynamic Properties of Materials at High Pressure

EVANS, William

Lawrence Livermore Natl Laboratory

CYNN, Hyunchae

Lawrence Livermore Natl Laboratory

LIPP, Magnus

Lawrence Livermore Natl Laboratory

We have developed a unique device, a dynamic diamond anvil cell (dDAC), which repetitively applies a time-dependent load/pressure profile to a sample. We are adapting this device to pulsed synchrotron radiation to time-resolve and take "snapshots" of pressure-induced transitions and phenomena. This capability allows studies of the kinetics of phase transitions and metastable phases at compression strain rates of up to 500 GPa/sec (~0.16 s-1 for a metal). Our approach adapts electromechanical piezoelectric actuators to a conventional diamond anvil cell design, which enables precise specification and control of a time-dependent applied load/pressure. This capability addresses the sparsely studied regime of dynamic phenomena between static research and dynamic shock-driven experiments. We present an overview of our work and experimental measurements that can be made with this device.

This work performed under the auspices of the US DOE by LLNL under Contract DE-AC52-07NA27344. HPCAT use is supported by DOE-BES, DOE-NNSA, NSF, and the W.M. Keck Foundation. APS is supported by DOE-BES, under Contract No. DE-AC02-06CH11357.

Brillouin Scattering at High Pressures and Temperatures: Potential Applications at a 3rd Generation Synchrotron Source such as PETRA III

SPEZIALE, Sergio
Deutsches GeoForschungsZentrum Potsdam
REICHMANN, Hans Josef
Deutsches GeoForschungsZentrum Potsdam

Brillouin spectroscopy is a technique that allows us to measure the shear elastic properties of solids at very high pressures and temperatures relevant for interpreting seismic heterogeneity in the deep Earth. In particular, Brillouin spectroscopy is one of the very few methods that can allow us to determine the full single-crystal elastic tensor of solids up to extreme conditions, with applications to the interpretation anisotropy from large scale seismic data. In addition to this, Brillouin scattering has a potential for the study of the equation of state of fluids at high pressures and temperatures. It allows us the determination of density by integration of the bulk modulus as a function of pressure. The determination of the density of complex amorphous solids is another relevant issue that could be addressed by Brillouin scattering, in combination with more demanding experimental techniques and computations that can offer complementary fine-structure information.

We propose a strategy for effectively integrating off-line Brillouin scattering measurements, X-ray diffraction and X-ray inelastic scattering at a 3rd generation synchrotron source. For instance, by combining Brillouin scattering and synchrotron X-ray diffraction in both axial and radial geometry we can develop a powerful tool for the study of the elastic behavior of unquenchable high-pressure solids synthesized as polycrystals, whose Brillouin scattering signal can only be interpreted based on the precise characterization of lattice-preferred-orientation.

Together with a range of applications of Brillouin scattering and synchrotron techniques at extreme conditions, we will also discuss perspectives about future technical developments to the current experimental design for high-pressure high-temperature Brillouin scattering in the diamond-anvil cell.

Plastic properties of mineral at high-pressure and –temperatures

MERKEL, Sebastien

LSPES - CNRS - Université Lille 1

In the last few years, a new experimental technique was designed to study the plastic properties of materials under high pressure in the diamond anvil cell. In those experiments, a polycrystalline sample is deformed between the two diamond anvils. Stress and lattice preferred orientations are then studied using diffraction in a radial geometry (i.e. with the incoming x-ray beam perpendicular to the load axis).

Those measurements are then compared with results of self-consistent elasto- and visco-plastic calculations in order to identify and constrain the plastic deformation mechanisms activated in the sample.

In this presentation, I will illustrate the technique and the results obtained with recent experiments and calculations. I will also describe new technical developments that allow us to perform experiments at temperatures on the order of 1200K.

Poster presentations

X-ray diffraction of weak scattering materials at megabar pressures

EREMETS, Mikhail

Max Planck Institute for Chemistry

TROJAN, Ivan

Max Planck Institute for Chemistry

MEDVEDEV, Sergij

Max Planck Institute for Chemistry

PALASYUK, Taras

Max Planck Institute for Chemistry

We will present recent results on X-ray diffraction in combination with optical and electrical studies of light elements: sodium [1], nitrogen [2] [3], and compounds with hydrogen and nitrogen: SiH_4 [4], HN_3, N_2H_4, LiN_3 and others at pressures up to about 200 GPa.

X-ray diffraction experiment with weak scatterers at multimegabar pressures is challenging, therefore it requires a special techniques which we are constantly developing. In particular, we use cBN/epoxy mixture as a gasket material [5]. It produces only a weak background and allows us to work with very small <10 μm sample required for multimegabar range. In addition, this gasket provides an enhanced thickness of the sample. We further significantly increased the sample volume in ~5 times with toroidal shaped diamond anvils [6].

1. Ma, Y., et al., Transparent Dense Sodium. Nature, 2009. 458: p. 182-185.
2. Eremets, M.I., A.G. Gavriliuk, and I.A. Trojan, Single-crystalline polymeric nitrogen. Appl. Phys. Lett. , 2007. 90: p. 171904.
3. Eremets, M.I., et al., Single-bonded cubic form of nitrogen. Nature Materials, 2004. 3: p. 558-563.
4. Eremets, M.I., et al., Superconductivity in Hydrogen Dominant Materials: Silane. Science 2008. 319: p. 1506-1509.
5. Eremets, M.I., et al., Superconductivity in boron. Science, 2001. 293: p. 272-274.
6. Trojan, I.A., et al., Transformation of molecular to polymeric nitrogen at high pressures and temperatures. In situ X-ray diffraction studies. Appl. Phys. Lett. , 2008. 93: p. 091907.

High-pressure crystallisation from solution: extending the search space for novel physical forms of "large small molecules"

FABBIANI, Francesca

Georg-August Universität Göttingen

FLORENCE, Alastair

University of Strathclyde

KUHS, Werner

Georg-August Universität Göttingen

SHANKLAND, Norman

University of Strathclyde

SOWA, Heidrun

Georg-August Universität Göttingen

The application of high pressure is a powerful method for exploring the polymorphic behaviour of simple molecular compounds.[1-4] Direct compression of either single crystals or powders, and crystal growth from the melt are two methods that have been successfully used to prepare new polymorphs.[1-4] The recent development of the experimental technique for in situ high-pressure crystallisation of single crystals from solution has allowed a wider range of compounds to be studied, including small-molecule pharmaceuticals, and has enabled the preparation of new forms at modest pressured, typically 0.1-1.5 GPa.[5] Exemplified with the study of the antibiotic ciprofloxacin,[6] whose chemical formula comprises 24 non-H atoms, we describe how: a) the technique can be extended for the study of larger, more complex compounds of biological importance; b) high-pressure forms can be recovered to ambient-pressure conditions. The key role played by the use of synchrotron radiation for collecting high-quality high-pressure data on single crystals of light-scattering organic compounds is also discussed.

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Ferroic materials at high pressures

GRZECHNIK, Andrzej

Condensed Matter Physics, Univ. Pais Vasco

FRIESE, Karen

Condensed Matter Physics, Univ. Pais Vasco

In this contribution, we will review our single-crystal studies of various (multi)ferroic materials as a function of pressure and temperature.

Ferroic phase transitions are characterized by the reduction of crystal symmetry very often associated with the appearance of a twin domain structure and/or an incommensurate structure. In addition, the low-symmetry phase usually shows a pronounced pseudo-symmetry with respect to the higher symmetry phase. Twinning, incommensurability, and high pseudo-symmetry could thus be understood as an indication of the existence of structural instabilities in the material under certain temperature and/or pressure conditions.

The transitions involving a symmetry reduction within the same diffraction class, which are not accompanied by the appearance of additional reflections, are very difficult to detect as they are exclusively reflected in changes of intensities in the diffraction diagrams. Observations of such transitions in high-pressure powder diffraction experiments have rarely been reported. Single-crystal diffraction, however, is suitable for such cases provided the data are of sufficient quality. An example is our study of the pressure-induced phase transitions in mixed valence vanadates NaV_6O_{11} and BaV_6O_{11}.

Towards Mbar-pressures at deep Earth's interior representing samples - the challenge of new light source based high pressure techniques for process research of the next decade and beyond

MUELLER, Hans J.
GFZ German Research Centre for Geosciences
LATHE, Christian
GFZ German Research Centre for Geosciences

The Earth's mantle has a mass of about 4.08 x 10^21 tons and represents 68 % of the total mass of the Earth. The Earth's mantle is only accessible by indirect methods, first of all seismological studies. The interpretation of these seismic data from the Earth's deep interior requires measurements of the elastic and inelastic properties of Earth materials under experimental simulated mantle and core-mantle boundary (CMB) conditions, i.e. conditions representative for Earth's deep interior. New seismological techniques based on the dense global station network resulted in fundamental improved knowledge about deep structures, as subduction down to the CMB, accumulation of low viscous material above the CMB, detection of rising plumes etc. One fundamental lesson from that is - contrary to classical imaginations deep Earth is just the opposite of being simple and stratified. There are clear relations between the Earth's interior and surface and atmospheric processes as climat development. The investigation of complex processes requires, technically speaking, strong controlling of gradients and assuring of minimum dimensions to truly represent natural conditions. There is also a strong indication of widespread unquenchable phases at extreme conditions requiring necessarily in situ observations. Recent geophysics sets of the tasks, recent material sciences and engineering delivers the tools, recent light source based mineral physics supplies recent numeric modeling with indispensable data to solve the geodynamic problems. The paper presents and summarizes some innovative high pressure techniques developed or under development for geoscientific experiments at light sources worldwide, including the techniques and results of the corresponding author's experiments at DESY beamlines.

Effect of Ba on pressure-induced structural changes in the relaxor ferroelectric PST

WELSCH, Anna-Maria
Mineralogisch-Petrographisches Institut, Universität Hamburg
MIHAILOVA, Boriana
Mineralogisch-Petrographisches Institut, Universität Hamburg
MAIER, Bernd
Mineralogisch-Petrographisches Institut, Universität Hamburg
ANGEL, Ross
Virginia Tech Crystallography Lab, Department of Geosciences, Blacksburg, USA
PAULMANN, Carsten
Mineralogisch-Petrographisches Institut, Universität Hamburg
GOSPODINOV, Marin
Institute of Solid State Physics, Bulgarian Academy of Sciences, Bulgaria
FRIEDRICH, Alexandra
Institut für Geowissenschaften, Universität Frankfurt
STOSCH, Rainer
PTB, Braunschweig
GÜTTLER, Bernd
PTB, Braunschweig
BISMAYER, Ulrich
Mineralogisch-Petrographisches Institut, Universität Hamburg

In strong contrast to $PbSc_{0.5}Ta_{0.5}O_3$ (PST), $Pb_{0.78}Ba_{0.22}Sc_{0.5}Ta_{0.5}O_3$ (PBST) exhibits canonical relaxor ferroelectric behaviour at low temperatures. This indicated that substitution of Pb by Ba in perovskite-type model ABO_3 structure induced significant nano-scale changes. The exact effects of Ba-doping under high pressures were investigated by in-house and synchrotron single-crystal XRD and Raman spectroscopy up to 10 GPa. Our results indicate that the stereochemically inactive Ba with no affinity to form lone pair electrons significantly broadens structural transformations over a pressure-range, without exhibiting a characteristic

critical pressure point. Elastic fields introduced by the size of Ba-cations have a significant role in the suppression of long-range ferroelectric ordering.