2nd Workshop for Extreme Conditions Research in a Large Volume Press at PETRA III

Abstracts book

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Detailed Program

2nd workshop for Extreme Conditions Research in a Large Volume Press at PETRA III

At Seminar room 456, Bldg. 25f, DESY, Hamburg, Germany
September 10-11, 2012

Program of Events

Monday, September 10 (Day1):

11:30 - 13:00 Registration

13:00 - 13:15, **Edgar Weckert** (DESY, Germany)
Welcome

13:15 - 13:30, **Wolfgang Drube** (DESY, Germany)
PETRA III extension

13:30 - 14:00, **Yanbin Wang** (GSECARS, University of Chicago, USA) ***invited***
Large-volume high-pressure research with synchrotron radiation for in-situ studies of earth and planetary materials

14:00 - 14:15, **David Dobson** (University College London, UK)
Material Properties measurements using synchrotron X-radiography

14:15 - 14:30, **Nadege Hilairet** (CNRS, Universite Lille 1, France)
Aseismic Deformation of Dehydrating Serpentinite

14:30 - 14:45, **Paul Raterron** (CNRS, Universite Lille 1, France)
Deformation of Earth Mantle Materials in the D-DIA Coupled with Synchrotron Radiation

14:45 - 15:15, coffee break

15:15 - 15:45, **Tetsuo Irifune** (GRC, Ehime University, Japan) ***invited***
Recent activities of Ehime group at BL04B1, SPring-8

15:45 - 16:00, **Hans J. Mueller** (Deutsces GFZ, Germany)
Measuring Elastic and Inelastic Properties under Simulated Earth's Mantle Conditions in Conjunction with Synchrotron X-Radiation

16:00 - 16:15**, Natalia Dubrovinskia** (University of Bayreuth, Germany)
LVP technique for synthesis and investigations of novel materials

16:15 - 16:30, **Marcus Schwarz** (TU-Bergakademie Freiberg, Germany)
Advanced Ceramics and New Assemblies with low X-ray Absorption for High Pressure-High Temperature Multianvil Synchrotron

16:30 - 16:45, **Norimasa Nishiyama** (DESY, Germany)
Synthesis of nanopolycrystalline stishovite using Kawai-type multi-anvil apparatus

17:00 - 18:00, poster presentations and coffee break (ground floor, 25f)

19:00 - 22:00, Workshop dinner (DESY bistro)

Tuesday, September 11(Day2):

08:55 - 09:00, Announcement

09:00 - 09:15, **Masashi Hasegawa** (Nagoya University, Japan) ***invited***
Synthesis and crystal growth of novel materials in high pressure and temperature using diamond anvil cell and laser heating

09:15 - 09:30, **Tomoo Katsura** (BGI, University of Bayreuth, Germany)
Possible high-energy X-ray diffraction studies at the LVP beam line in PETRA III Extension

09:30 - 09:45, **Simon Hunt** (University College London, UK)
X-radiography based method for measuring thermal diffusivity under in-situ conditions

09:45 - 10:00, **Chrystele Sanloup** (University of Edinburgh, UK)
Structure and density of silicate melts using large-volume presses

10:00 - 10:15, **Oliver Beermann** (Universitat Kiel, Germany)
From high to moderate p/T-conditions - in situ insights to core formation scenarios as well as to magmatic and volcanic processes

10:15 - 10:45, coffee break

10:45 - 11:00, **Norimasa Nishiyama** (DESY, Germany) beamline, LVP equipment, detectors

11:00 - 12:00, Discussion

Poster presentations

Fabrice Brunet (ISTERRE - CNRS, France)
Hanns-Peter Liermann (DESY, Germany)
Tony Bell (DESY, Germany)
Norimasa Nishiyama (DESY, Germany)

Large-volume high-pressure research with synchrotron radiation for in-situ studies of earth and planetary materials

WANG, Yanbin (Center for Advanced Radiation Sources, Univ. Chicago) (***INVITED***)

The field of high-pressure earth and planetary sciences has changed dramatically overthe past few decades, primarily owing to the increasingly sophisticated techniquesthat are constantly being developed. Capabilities of modern synchrotron and neutron sources provide enormous opportunities for new types of experimentation at highpressure (P) and temperature (T). Accordingly, many new high-pressure devices havebeen developed to take advantage of these advances. This presentation will focus on a range of new developments for characterizing physical and chemical properties of minerals and rocks at simultaneous high P and T, using the large-volume presses (LVP). Particularly, we will discuss new developments at GSECARS of the Advanced Photon Source for the following applications: (1) structures of crystals and liquids, (2) Elasticity of solids and potential applications for liquids, (3) rheological properties of minerals (poly- and single crystals) and melts, and (4) acoustic emission experiments coupled with x-ray diffraction and imaging to evaluate hypotheses on deep-focus earthquakes. In addition, high-pressure 3D tomographic imaging technique will be introduced, which has been used to study various phenomena, including (1) texture development in multi-phase materials under shear deformation,(2) segregation of Fe-rich melts from silicate, and (3) equations of state of melts.

Future prospects will be discussed base on a few new initiatives.

Material Properties measurements using synchrotron X-radiography

DOBSON, David (University College London)
SIMON, Hunt (University College London)

X-radiography is perhaps the simplest use of synchrotron X-rays. Despite (or perhaps because of) this, it remains a powerful method to probe the state of matter under extreme conditions. It has been used to measure the (1) liquid viscosity, (2) density, (3) rheology and (4) thermal diffusivity of samples in multi-anvil and other solid-media presses.

1) Liquid viscosity is measured from Stoke's equation of a falling sphere, using X-radiography to image the passage of the sphere. If spheres with different densities are used the equations can be solved for density and viscosity of the liquid, but densities thus derived tend not to be very accurate.

2) Similarly to (1)density can be measured by imaging the passage of an inert sphere through the liquid if the sphere has similar density to the liquid. By following the P-T conditions where the sphere floats or sinks an isograd can be mapped out where the liquid and sphere have identical densities. Alternatively, if monochromatic beam is available the absorption contrast between the sample and a reference material can be measured and converted into a density via the Beer-Lambert law. Neither of these density measurements are as precise as X-ray diffraction methods but they can be used on non-diffracting materials such as geophysically-important fluids and melts.

3) Rheology is determined by imaging the change in length of a sample under a controlled non-hydrostatic stress.

4) We have recently developed a radiographic Angstrom method to measure the thermal diffusicity. In this technique the temperature in a cylindrical furnace is varied sinusoidally and the resultant thermal expansion of the sample is imaged as a proxy for temperature. X-radiography of marker foils placed appropriately in the sample allows the phase angle to be determined at all radii.

We will discuss the requirements of beam characteristics, imaging optics and press
control systems to make these measurements using synchrotron X-radiography.

Aseismic Deformation of Dehydrating Serpentinite

GASC, Julien (CARS The University of Chicago)
HILAIRET, Nadege (CNRS - Université Lille 1)
WANG, Yanbin (CARS The University of Chicago)
SCHUBNEL, Alexandre (CNRS, ENS)

Serpentinite is one of the main constituents of the subducting slabs. Its dehydration is believed to be responsible for triggering earthquakes at intermediate depths (i.e., 60-300 km). Based on experimental results, some authors have proposed mechanisms that explain how brittle deformation can occur despite high pressure and temperature conditions. A recent study showed that, even for fast dehydration kinetics, ductile deformation could take place rather than brittle faulting in the sample (Gasc et al, 2011). This latter study was conducted in a multi-anvil apparatus without the ability to control differential stress during dehydration.

We have since conducted controlled deformation experiments in the deformation-DIA (D-DIA) on natural serpentinite samples at sector 13 (GSECARS) of the APS. Monochromatic radiation was used to collect, full diffraction rings and radiographies of the samples. which allow us to determine stress and the strain of the sample during the deformation. An Acoustic Emission (AE) recording setup was used to monitor microseismicity from the sample, using piezo-ceramic transducers glued on the basal truncation of the anvils.

The samples were deformed at strain rates of 10–5-10–4 s–1 under confining pressures of 3-5 GPa. Dehydration was triggered during the deformation by heating the samples at rates ranging from 5 to 60 K/min. Before the onset of the dehydration, X-ray diffraction data showed significant amounts of stress which plummeted when dehydration occurred. No AE events accompanied this stress drop suggesting ductile deformation of the samples. Hence, unlike many previous studies, we document here an “aseismic” interplay between deformation and dehydration of natural serpentinite.
These results suggest dehydration of serpentinite alone cannot be responsible for intermediate earthquakes.

Deformation of Earth Mantle Materials in the D-DIA Coupled with Synchrotron Radiation

RATERRON, Paul (CNRS / Université Lille 1, France)

The past decade has brought numerous technical and theoretical developments allowing new approaches in the investigation of materials plasticity at high pressure. Highpressure devices set at synchrotron facilities, such as the Deformation-DIA apparatus (D-DIA) - a multi-anvil apparatus with cubic geometry (Wang et al., 2003, Rev. Sci.Instrum., 14, 3002) - allow now quantifying materials rheology at pressure (P) in excess of 10 GPa (e.g., Raterron & Merkel, 2009, J. Synchrotron Rad., 16, 748). In the D-DIA, deformation is promoted by moving forward top and bottom anvils (mounted on individual inner rams) while allowing the lateral anvils to retract by maintaining constant the main-ram oil pressure. This promotes specimen axisymmetric compression at constant P and temperature (T). In the D-DIA coupled with synchrotron radiation, the applied stress is measured from diffracted X-rays arising from polycrystalline materials of known elastic properties placed in the compression column, and specimen strain rate are measured by time-resolved radiography.

The high-pressure rheological properties of Earth upper-mantle materials (olivine, pyroxenes, etc.) were investigated using the D-DIA at the X17B2 beamline of the National Synchrotron Light Source (NSLS, NY, USA). Aggregates, as well as single crystals, have been deformed at P and T conditions relevant to the upper mantle. The obtained rheological laws, while integrated into analytical or computational modeling, shade new light on upper mantle plasticity (e.g., Castelnau et al., Comptes Rendus Physique, 11, 304-315, 2010; Raterron et al., PEPI, 200-201, 105-112, 2012).

It will be shown that combining state-of-the-art high-P deformation experiments with aggregate deformation computations allows addressing part of the complexity of uppermantle thermal convection. (Paul.Raterron@univ-lille1.fr)

Recent activities of Ehime group at BL04B1, SPring-8

IRIFUNE, Tetsuo (Geodynamics Research Center (GRC), Ehime University) (***INVITED***)

The LVP beamline, BL04B1, was constructed in 1997 as one of 10 preceding beamlines among some 60 beamlines currently available at SPring-8. The GRC group of Ehime University has been playing important roles in construction of the beamline, updating and maintenance of instruments, training new users, etc., in addition to pioneer studies in high-pressure mineral physics. Consequently, the group has been appointed as one of 7 Power Users (PUs) amongst hundreds of research groups using SPring-8 beamlines. Here, I will review some recent activities of the GRC group at the LVP beamline, focusing on technical developments relevant to mineral physics studies, which can be categorized as 1) higher pressure generation using sintered diamond and nano-polycrystalline diamond (NPD or "Hime diamond") anvils, 2) deformation of minerals in a large D-DIA assembly, and 3) sound velocity measurement using ultrasonic technique. Based on these latest techniques, we have been studying precise determinations of phase transitions, P-V-T relations, rheological and elastic properties of major high-pressure minerals/rocks, particularly focusing on those under P-T conditions of the mantle transition region (depths of 410-660 km, equivalent to 13-24 GPa in pressure) and the lower mantle (660-2900 km; 24-136 GPa). Aspects of the outcomes relevant to deep Earth mineralogy are also to be discussed.

Measuring Elastic and Inelastic Properties under Simulated Earth's Mantle Conditions in Conjunction with Synchrotron X-Radiation

MUELLER, Hans J. (Helmholtz Centre Potsdam, GFZ German Research Centre for Geosciences)
LATEE, Christian (Helmholtz Centre Potsdam, GFZ German Research Centre for Geosciences)

The Earth's deep interior is only accessible by indirect methods, first and foremost seismological studies. The interpretation of these seismic data and the corresponding numerical modelling requires measurements of the elastic properties of representative Earth materials under experimental simulated in-situ pressure-temperature conditions. For experiments under uppermost mantle conditions highly metamorphic and exhumed natural rocks can still be used as specimen. The benefit of considering a natural texture is attended by the possible influence of minor retrograde metamorphosis.

The simulation of transition zone conditions and beyond require multi-anvil devices, also called as large volume presses, because they provide sample volumes 3 to 7 orders of magnitude bigger than in diamond anvil cells. The sample size of several cubic millimeters allows elastic wave frequencies in the low to medium MHz range. Together with the small and even adjustable temperature gradients over the whole sample this technique makes anisotropy and grain boundary effects in complex systems accessible for elastic and inelastic properties measurements in principle. The measurements of both elastic wave velocities have also no limits for opaque and encapsulated samples. The application of triple-mode transducers and the data transfer function technique for the ultrasonic interferometry reduces the time for saving the data during the experiment to about a minute or less. That makes real transient measurements under non-equilibrium conditions possible. A further benefit is, both elastic wave velocities are measured exactly at the same time. Under high pressure conditions the influence of sample deformation is so important that ultrasonic interferometry requires the exact sample deformation measurement under in situ conditions using synchrotron radiation. Performing measurements with molten samples require encapsulation. We developed a special sample encapsulation for ultrasonic interferometry which do not interfere the acoustic measurements. Various experimental techniques and their results are described including standard-free pressure measurement, elastic properties across unquenchable phase transitions, measurements with molten encapsulated samples and falling sphere viscosimetry.

LVP technique for synthesis and investigations of novel materials

DUBROVINSKAIA, Natalia (Material Physics and Technology at Extreme Conditions, Laboratory of Crystallography, University of Bayreuth)
DUBROVINSKY, Leonid (Bayerisches Geoinstitut, University of Bayreuth)
GOU, Huiyang (University of Bayreuth)
PARAKHONSKIY, Gleb (University of Bayreuth)
BYKOVA, Elena (University of Bayreuth)

Exploration and development of novel, in particular superhard, materials is a highly multidisciplinary scientific field with a great potential for applications. Due to the synergy of the large volume press (LVP) and diamond anvil cell (DAC) techniques, as well as applying modern physical, chemical and engineering methods of investigations of material properties, we obtained and studied a series of hard and superhard materials in boron, carbon, and related systems. Their physical properties have been shown to be influenced not only by their chemistry and structure, but also by the degree of their crystallinity in terms of grain size, grain boundary structure and the composite nature of nano- and polycrystalline aggregates. It was demonstrated that by tuning these factors, there is a great potential to vary technologically important thermoelastic and mechanical properties of superhard materials such as hardness, fracture toughness, thermal stability and wear resistance.
A number of novel high pressure materials in the pure boron system including newly synthesised metastable phases, as well as a number of borides will be presented.

Prospective advantages of in situ studies using the online Large Volume Press at PETRA III will be outlined.

Advanced Ceramics and New Assemblies with low X-ray Absorption for High Pressure-High Temperature Multianvil Synchrotron

SCHWARZ, Marcus (Freiberg High Pressure Research Centre, Institute for Inorganic Chemistry)
BARSUKOVA, Tatiana (Freiberg High Pressure Research Centre, Institute for Inorganic Chemistry, TU-Bergakademie Freiberg)
SCHIMPF, Christian (Freiberg High Pressure Research Centre, Institute for Material Science, TU-Bergakademie Freiberg)
LI, Guan (Freiberg High Pressure Research Centre, Institute for Inorganic Chemistry, TU-Bergakademie Freiberg)
RAFAJA, David (Institute for Material Science, TU-Bergakademie Freiberg); Prof. KROKE, Edwin (Institute for Inoranic Chemistry, TU-Bergakademie Freiberg)

In a number of visits at the MAX200X multianvil X-ray diffractometer at Beamline W2 of the DORIS source at Hasylab, DESY, we have constructed and tested a number of different designs for the sample assembly, including heater design, X-ray transparent and high temperature resistant amorphous window materials and new types of gaskets with low absorption. The Freiberg High Pressure Research Centre has thus established capabilities for home-fabrication of the majority of the parts for octahedral pressure cells, including octahedra and insulator tubes.

Utilization of the polymer-derived ceramic (PDC) technique in addition to the oxide ceramics traditionally used for multianvil experiments, such as MgO, ZrO2 and Alumina, proved to be particularly useful. Polysilsesquioxane resin can be effectively used as an active binder to fabricate ZrO2 insulator tubes and MgOoctahedra with firing temperatures as low as 900°C, without compromising mechanical properties. Pyrolysis of polyborosilazane yields an amorphous Si/B/C/N-ceramic, which shows excellent performance as a highly heat-resistant and thermally insulating material for X-ray beam windows. We also show first results on our attempts to fabricate precursor-based TiC-SiC heaters that are directly painted on the ZrO2
insulator tubes.

In the higher pressure region, where the gaskets are considerably squeezed and the beam-path through the absorbing medium is long, absorption effects in conventional gasket materials like pyrophyllite become eminent. This is especially critical if fast transient processes are to be monitored with desirably short data collection times. A composite gasket material with improved X-ray transmission was developed and successfully applied. It is based on a layered, aromatic carbon nitride (amide) material and shows similar mechanical characteristics as pyrophyllite. We believe that our experiences and developments could be beneficial for future concepts at DESY as well as other synchrotron sources.

Synthesis of nanopolycrystalline stishovite using Kawai-type multi-anvil appratus

NISHIYAMA, Norimasa (DESY, FS-DO)

Stishovite is a high pressure polymorph of SiO2 stable at pressure above 9 GPa. It has rutile structure where silicon atoms occupy the centers of SiO6 octahedra. It is a very dense mineral which is more than 60% denser than quartz and is elastically extremely stiff (the bulk modulus is 316 GPa). It has been known that stishovite is the second hardest oxide next to the cotunnite-type TiO2. A previous study reported that a value of fracture toughness of single crystal stishovite is 1.6 MPa m1/2, which is lower than that of single crystal corundum. Stishovite single crystal is very brittle. There has been no report on fracture toughness of polycrystalline stishovite.

We synthesized polycrystalline stishovite at 15 GPa and at temperatures between 1273 and 2073 K. Nanocrystalline stishovite samples were obtained between 1273 and 1673 K. The average grain size increases with synthesis temperature: 50 nm at 1273 K and 240 nm at 1673 K. Samples synthesized at 1873 and 2073 K have microcrystalline grains. We performed Vickers indentation tests for the samples synthesized between 1273 and 1873 K to determine hardness and fracture toughness. The determined values of Vickers hardness were almost constant at about 29 GPa. This value is consistent with those reported in previous studies including some recent theoretical calculations. The values of fracture toughness of the samples synthesized at 1273 and 1473 K were 13 MPa m1/2, which is more than 8 times higher than that of single crystal stishovite. Nanopolycrystalline stishovite is significantly toughened. Above 1473 K, fracture toughness decreases with synthesis temperature.

Nanopolycrystalline stishovite has potential to be used for industrial purposes because of simultaneous very-high hardness and toughness.

Synthesis and crystal growth of novel materials in high pressure and temperature using diamond anvil cell and laser heating

HASEGAWA, Masashi (Department of Crystalline Materials Science, Nagoya University) (***INVITED***)
NIWA, Ken (Department of Crystalline Materials Science, Nagoya University)
KUSABA, Keiji (Department of Crystalline Materials Science, Nagoya University)

Synthesis and crystal growth of nanostructured materials in high pressure and high temperature over ~GPa and ~2000 K, especially over 10 GPa is one of the next new region of the material science. It is attractive from the viewpoint of not only the geoscience but also the crystal chemistry, crystal growth and materials science. The diamond anvil cell (DAC) is a hand-size small instrument and provides us easy means to achieve the conditions over several GPa pressure range. It can be combined easily with the infra-red laser heating system to get higher temperature of a sample over ~2000 K. This is the LASER-DAC high pressure high temperature system. The system also provides us supercritical fluid at high temperature and high pressure over ~GPa as a solvent for the chemical reaction and crystal growth, such as nitrogen, oxygen, carbon dioxide and so on. In this talk, we will report and discuss synthesis and crystal growth of various unique materials such as the rutile-type oxide hollow tube crystals, boron compound icosahedron crystals and metal nitride crystals, etc. using the LASER-DAC. The comparison between LASER-DAC and Large-size Press are also discussed from the viewpoints of chemistry and materials science related to our researches.

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Possible high-energy X-ray diffraction studies at the LVP beam line in PETRA III Extension

KATSURA, Tomoo (Universität Bayreuth)
FROST, Dan (Universität Bayreuth)

1) Compositional dependence of thermoelastic properties of lower mantle perovskite Although thermoelastic properties of $MgSiO_3$ perovskite have been extensively investigated, those of more realistic composition are poorly understood. We will directly compare equations of states of $MgSiO_3$ and Fe-Al bearing perovskites by means of angle-dispersive X-ray diffraction with sintered diamond anvils up to 60 GPa.

2) Dislocation recovery experiment by means of Bragg diffraction imaging Bragg diffraction imaging is a method to observe dislocations in a crystal. In the present plan, we will load two forsterite single crystals, one of which is almost perfect, and the other contains high density dislocation. We will compare the intensity of the same Laue spots between these two crystals. We will heat the crystals to certain temperature to anneal them and observe change in intensity of diffraction image. From these observations, we will estimate dislocation mobility as a function of pressure and temperature.

3) Observation of single crystal growth by means of Bragg diffraction imaging. We will load one single crystal in a cell of single crystal growth. We will observe growth of the crystal and their defect structure by means of Bragg diffraction imaging. From this observation, we will obtain the mechanism of single crystal growth and find the best procedure to obtain high-quality single crystals of high pressure minerals.

4) Phase relations

The phase relations of possible lower-mantle phases at pressures above 25 GPa have been studied by using DAC. The phase relations between 25 and 60 GPa will be studied using sintered diamond anvils. The possible target reactions are as follows: perovskite-corundum phases in $MgSiO_3$-Al_2O_3, Calcium ferrite phases in $NaAlSiO_4$-$MgAl_2O_4$., distortion of stishovite in SiO_2-$AlHO_2$

X-radiography based method for measuring thermal diffusivity under in-situ conditions

HUNT, Simon (University College London)
DOBSON, David (University College London)
LI, Li (Mineral Physics Institute, Stony Brook University)
WEIDNER, Don (Mineral Physics Institute, Stony Brook University)

We have recently developed a variation of the Angstrom method for measuring thermal diffusivity at high pressures by using X-radiography.

The Angstrom method for measuring thermal diffusivity at high pressure uses a stationary thermal wave which is induced in the sample by varying the power sinusoidally in the surrounding cylindrical furnace. The thermal diffusivity (κ) is determined from the phase lag, Φ0 - ΦR, and amplitude difference, θ0/θR, of the thermal wave between points at the axis of the sample and radius, R (e.g. Khedari et al., 1995).
Our method differs from previous multi-anvil implementations of the Angstrom method in that instead of using thermocouples to monitor the temperature variation we use thin strips of metal foil, which are placed at discrete intervals along the sample length and imaged X-radiographically. The metal strips monitor the thermal expansion of a slice across the sample in response to the sinusoidal temperature profile. This represents an improvement over previous methods since (i) the change in temperature is averaged along the sample length, (ii) we measure the phase of the thermal wave at all radii and (iii) since the expansion of the sample is observed as a proxy for the change in temperature there are no problems associated with contact thermal resistance at the thermocouples. Furthermore, this development does away with the need to prepare long cylinders of weakly metastable phases with a thermocouple inserted precisely down the middle; a process that is technically challenging.

To date we have measured the thermal diffusivity of NaCl, olivine, pyroxenes, majorite and a number of other upper-mantle and transition-zone phases. The measurements we have made are all in agreement with previously published data. We therefore promote this method as a significant development over previous Angstrom methods and call for the capabilities required to perform this type of experiment in the Large Volume Press beamline at PETRA-III.

Structure and density of silicate melts using large-volume presses

SANLOUP, Chrystele (University of Edinburgh)

In situ studies of silicate liquids at high pressure aim at answering questions relevant to the presence of magmas at depth, whether that be in the present Earth or in its earliest times, during differentiation of the planet. Melts have unique physical and chemical properties, which vary as a function of temperature (T), pressure (P), and chemical composition. Understanding the macroscopical physical properties of magmas requires an accurate microscopic structural description.

However, only a limited amount of information exists about changes in silicate melts structure with T or P or both, and that information is mostly indirect as it comes from the study of glasses. Pioneering in situ structural data have been obtained by japanese groups, however they are limited to less than 10 GPa, a pressure range too low for any significant structural changes in silicate melts to be observed. That blurrs out our understanding of mineral/melt partitioning, and hence of partial melting.

The LVP beamline in PetraIII offers a unique opportunity to look at the structure of magmas (i.e. main ion- ion distances and respective coordination numbers) at very high pressure. I will present structural data obtained on silicate melts using the Paris-Edinburgh press combined with either angle-dispersive or energy-dispersive x-ray diffraction set-ups, and show how we can measure the compressibility of melts.

The pros and cons of both techniques for the study of silicate melts will be discussed.

From high to moderate p/T-conditions – in situ insights to core formation scenarios as well as to magmatic and volcanic processes

BEERMANN, Oliver (CAU Kiel)
HOLZHEID, Astrid (CAU Kiel)
KEGLER, Philip (CAU Kiel)

The knowledge of phase chemistries at phase equilibria at high or moderate p/T-conditions is fundamental to transfer experimental data to natural geologic systems. For example, partitioning coefficients between silicate melt phase and metallic as well as sulfide melt phases are important to understand fractionation processes controlling the chemical evolution of the earth. But sulfide melt phases cannot be quenched into a single phase due to immiscibility between the Fe-rich component and stoichiometric FeS. Some metallic melt phases can also not be quenched into a single phase and characteristic exsolution lamellae form during quench. It is therefore necessary to perform in situ measurements at the experimental p/T-conditions to obtain the equilibrium phase chemistry and thus derive reliable partitioning data.

Another example of the necessity to determine chemical compositions of coexisting phases at high or moderate p/T-conditions are in situ chemical analyses of geological relevant fluids. This would not only allow getting solubility data, also information about the reaction kinetics can be obtained. Those data are necessary to understand elemental transport mechanisms caused by subduction zone fluids finally released to the earth surface through subduction zone volcanism or to understand the kinetics of ore formation processes caused by late stage magmatic hydrothermalism.

In addition to determine the phases' chemistry, while the phases of interest are still at the experimental p/T-conditions, in situ image analyses of for example bubble formation processes at the onset of magma degassing depending on the fluid's chemistry (e.g., effect of dissolved H2O, CO2, and S in the fluid on nucleation of bubbles) are very useful to predict the explosive potential of volcanic eruptions.

Study of the kinetics of phase transitions and mineral reactions at HP-HT: what for and how?

BRUNET, Fabrice (ISTERRE - CNRS)
GASC, Julien (GSECARS)
SCHUBNEL, Alexandre (ENS-CNRS)
BAGDASSAROV, Nikolai (GOETHE UNI-Frankfurt)
HETENYI, György (ETH-Zurich)
MUELLER, Hans-joachim (GFZ)
LATHE, Christian (Hasylab)

Phase transitions and mineralogical reactions can be viewed as relaxation phenomena which operate in rocks in response to a thermochemical disequilibrium imposed by changing geodynamical conditions (rock burial, heating, stress loading, chemical changes in open systems). Depending on their relaxation rate (reaction/transition kinetics), these transformations can impact several important geophysical properties(plate dynamics, faulting instability, heat flux). The dependency of reaction kinetics on temperature is well understood as a thermally activated process with Arrhenian behaviour. However, temperature changes in reactive processes also imply changes in reaction free energy, the effect of which on kinetics is much less understood. In most reactive systems, water acts as a catalyst which enhances grain boundary diffusivity; Dry systems are recognized as much less reactive as water saturated ones. However, the effect of water activity on reaction kinetics is far from being understood although it is recognized as a major controlling parameter. Grain boundary diffusion in a polycrystalline system is a function of grain boundary width and geometry, two parameters which are extremely difficult to determine insitu. Finally, the effect of deviatoric stress on mineral transformation kinetics has been mostly overlooked. Overall, there is a clear lack of kinetic data at HP-HT for reactive mineral assemblages, for dehydration reactions and for phase transitions.

In-situ X-ray powder diffraction is the obvious tool to address reaction kinetics.
Over the past years, we have been trying to develop complementary tools such as insitu
impedance spectroscopy or acoustic emission monitoring in order to complement XRPD data towards the characterization and the understanding of physico-chemical property variations (chemical diffusivity, grain boundary hydration, embrittlement) in reacting natural and analogous systems. These measurement techniques can be (and are) installed on LVP's.