Poster Presentations

Prediction and synthesis of polyhydrides in the Y-Fe-H system

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Computational studies have successfully predicted the dramatic uptake of hydrogen by metals under pressure leading to the formation of superhydrides. These new materials demonstrate exceptional properties, such as almost ambient temperature superconductivity, record hydrogen storage and superionicity, as exemplifies on the case of LaH10[1,2]. Up to now, most of the studies have been performed on binary hydrides in the 100 GPa range and none of the discovered superhydrides could be recovered at ambient pressure[3]. Hence, there is a great current interest to possibly form such H-rich compounds at moderate pressure and to recover them at ambient pressure by adding a third element.

We have undertaken a search for ternary super-hydrides in the Fe-Y-H system by coupling calculations and experiments. We have first focused our attention on the hydrogen uptake by the binary alloy YFe2. We have performed Crystal Structure Prediction (CSP) and DFT calculations on a ternary hydride YFe2Hx compound which showed that YFe2H7 should be synthesised by compressing YFe2 in excess hydrogen in the 25 -50 GPa range. Doing XRD at ESRF, we measured the synthesis of YFe2H7. The metastability of this compound was investigated on the release of pressure. A strong hysteresis between the uptake and the withdrawal is observed. Only YFeH5 is stable at ambient pressure. The first results on the existence of other poly-hydrides in the Y-Fe-H system will be discussed.

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High-pressure structural study of CeAl2

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Quantum criticality in strongly correlated electron systems provides a promising setting for discovering emergent quantum phenomena and novel quantum phases [1]. A Quantum Critical Point (QCP) arises when a material undergoes a continuous phase transition at zero Kelvin due to strong quantum fluctuations. These quantum fluctuations can be tuned using non-thermal control parameters such as magnetic field, chemical doping, or external pressure. Quantum criticality at the border of magnetism is interesting due to the possibility of unconventional superconductivity, non-Fermi liquid behaviour (NFL) and other emergent phenomena. In this project, magnetism is suppressed to quantum criticality using hydrostatic pressure to understand these ordered systems better and discover new phases that cannot be readily seen under ambient conditions. Application of pressure can change the density of the condensed matter and induce myriad changes in its physical and chemical properties. It's an excellent tool to tune the ground state of materials without introducing additional disorders.

One of the prominent examples of strongly correlated systems is the Cerium-based intermetallic heavy fermion compounds. Here we present the high-pressure structural studies of a trivalent rare-earth dialuminide, CeAl2. It crystallises in a cubic Laves phase (MgCu2 structure) with space group Fd-3m. CeAl2 orders into a spin density wave (SDW) antiferromagnet (AFM) at TN = 3.8 K, and it has a Kondo temperature, TK, of 6 K [2]. Field dependence studies of resistivity and Anomalous Hall Effect (AHE) indicate strong magnetic fluctuations and the formation of nanoscale spin-polarised regions in the vicinity of Ce centres, above TN [3-4]. The complex magnetic lattice with competing interactions and its unique magnetic and electronic properties makes CeAl2 an interesting candidate for quantum criticality studies. The competing interactions in CeAl2; RKKY and Kondo effect are sensitive to pressure, and the SDW AFM order slowly disappears with increasing pressure. The quantum critical point arises at a critical pressure of 2.7 GPa, with an accompanying NFL state [5]. Pressure dependence of magnetoresistivity and Hall coefficient asserts a crossover in the electronic state, from incoherent to coherent Kondo scattering regime [5-6]. To understand pressure-induced changes in physical properties and emergent phenomena near QCP, a detailed structural investigation of CeAl2 under high-pressure is essential. Structural studies of CeAl2 at variable temperatures were carried out in the pressure range of 0.9 – 14.1 GPa above 8K, whereas room temperature studies were done within 1.49 – 27.27 GPa. Structural and electronic changes, microstructural effects, and bulk modulus of the lattice under varying pressure are discussed and compared with the literature.

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Temperature and high-pressure studies of layered perovskite Cs[C(NH2)3]PbI4

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Metal halide perovskites are a promising class of materials for optoelectronic applications because of their broad light absorption ranges, tuneable bandgaps, long carrier lifetimes, and high photoluminescence quantum yields [1]. Considerable interest has also been focused on two-dimensional (2D) perovskites that have better stability than their three-dimensional counterparts. Here we present a comprehensive study of the effects of pressure and temperature on the structural and optical properties of CsC[(NH2)3]PbI4. This material is a 2D perovskite that crystallises in the orthorhombic system with the space group Pnnm[2]. The compound has a layered structure, where cesium and guanidinium cations are situated between the corner-shared PbI6 octahedra layers.

We performed differential scanning calorimetry in the temperature range 93–500 K and single-crystal X-ray diffraction measurements in the temperature range 200–380 K. The collected data demonstrate a second-order phase transition occurring at 320 K, where the space group changes from Pnnm to Ima2. In the low-temperature phase, the lattice parameters a and c increase with temperature, while b shows a negative thermal expansion. At the transition point the expansion of b changes to the positive one, and as a result all lattice parameters increase with temperature in the high-temperature phase. Our crystal structure determinations allow us to discuss the temperature-induced structural changes. Furthermore, we performed optical and structural measurements in a diamond anvil cell up to 4.5 GPa. Daphne oil 7575 was used as a hydrostatic medium. Optical studies show that at atmospheric pressure the absorption edge of CsC[(NH2)3]PbI4 is located at 558 nm and gradually redshifts upon compression. The structural measurements show that the lattice parameters continuously decrease under pressure and the crystal preserves the symmetry of space group Pnnm, indicating that the crystal structure is stable within the studied pressure range. This is untypical because metal halide perovskites usually undergo structural phase transitions under relatively low pressures.

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Sequential spin state transition and intermetallic charge transfer in PbCoO3 under high-pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Spin state transition and intermetallic charge transfer can both essentially change the d-electronic configurations without introducing any doping element, giving rise to drastic variations for materials structures and physical properties. However, these two effects are never found to occur together in a specific material. Here we report that pressure can sequentially induce the spin state transition and intermetallic charge transfer in PbCoO3 perovskite which possesses a very rare A- and B-site ordered charge format Pb2+Pb4+3Co2+2Co3+2O12 at ambient conditions. With increasing pressure, the high spin Co2+ gradually changes to be low spin, while the AA'3B2B'2O12-type crystal structure maintains unchanged with insulating conductivity below 20 GPa. Between 20 and 30 GPa, the ordered Co2+/Co3+charge states are melted into a mixed Co2.5+ at higher temperatures accompanying with metallization, but lowering temperature leads to a metal-insulator transition. Moreover, Pb4+-Co2+ intermetallic charge transfer also takes place in this pressure region. With further compression up to 30 GPa, the charge transfer completes, and the compound most probably develops into an A-site ordered Pb2+Pb4+Co3+2O6 phase. Since the B-site is fully occupied by the low spin Co3+, insulating behaviour emerges again in this phase.

Equation of State for Ice Ih with explicit contributions from proton-disorder and molecular defects

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Equation of State for Ice Ih with explicit contributions from proton-disorder and molecular defects.

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Recent neutron diffraction studies on ice Ih at ambient pressure and temperatures between 1.5 and 270 K improved the accuracy in the thermal expansion of the lattice parameters and the corresponding molecular volume by an order of magnitude[1] with respect to the latest reference data[2]. This provided the basis for an update of the equations of state for the full range of stability for ice Ih in terms of a thermodynamic model including contributions from phonons in quasi-harmonic and anharmonic approximation together with contributions from proton disorder and premelting effects [3]. A study of the effects of grain size and quenching on the lattice parameters [4] led to a better understanding of the contribution from proton disorder and the range of uncertainty introduced to the equations of state at low temperatures by quenching. A macroscopic dilatation study of single-crystalline ice Ih at ambient pressure and temperatures between 5.5 and 265 K resulted in a dilemma due to the observation of significantly smaller dilatations [5]. The differences in the dilatation of the a- and c-axis are illustrated in figures 1 and 2 for H2O and D2O ice Ih respectively. The reduction of the macroscopic change with respect to the neutron-diffraction data is almost linear above 80 K in both cases and slightly anisotropic in the case of D2O. Since only one sample of H2O and one for D2O was studies in the macroscopic dilatation measurements it is not clear if these small differences are significant. A small shift between H2O and D2O data seem to indicate only molecular defects could be responsible for these differences. The different contributions to the thermal expansion of H2O ice Ih and a comparison with the “reference data” [2] at ambient pressure and at 200 MPa (just below the phase transitions to ice II, III, IX) are represented in the figures 3 and 4, illustrating different effects of sample quality on the equation of state.

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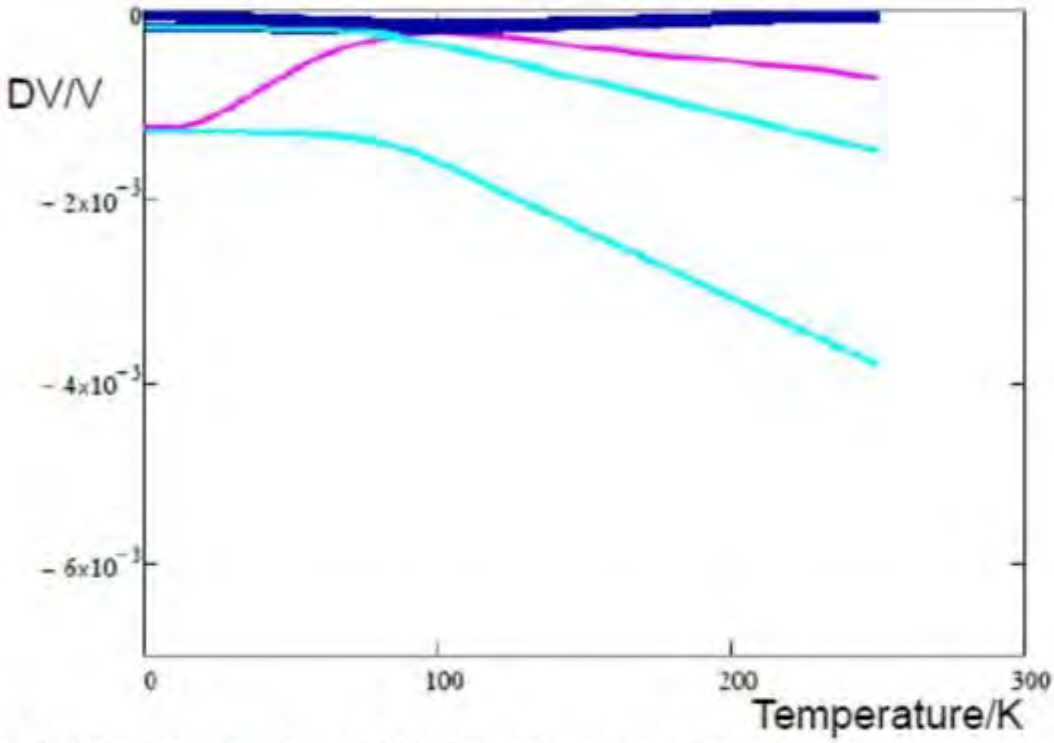

Figure 4: Relative contribution at 200 MPa of the proton disorder (dark blue range) and defect contribution (range between light blue the lines) to the volume expansion with respect to the phonon contribution and difference to the "reference data" (pink line)

The soft matter and chemistry support facilities at the Institut Laue-Langevin

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The Institut Laue-Langevin has been performing experiments at the cutting edge of research for more than 40 years now, in fields as varied as molecular biology, chemistry, fundamental physics, materials science and the environment. The ILL provides the scientific community with 40 high-performance instruments constantly maintained at the highest state of the art. To exploit the full potential of the high-performance instruments, a number of support facilities are available at the ILL, i.e., the deuteration laboratory, sample environment or the computing for science groups. In this contribution, we aim at presenting the support given by the chemistry laboratories and the partnership for soft condensed matter (PSCM). The chemistry facility at the ILL manages several wet chemistry labs, as well as chemistry laboratories fully equipped to handle nano-powders. The PSCM laboratories provide approx. 30 laboratory scale equipment aimed at the characterization and preparation of samples in the domain of soft condensed matter, including high-pressure equipment.

In situ and ex situ studies of silicate glasses and glass-ceramics under high-pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Glasses are defined as amorphous solids obtained from melting-quenching, resulting in isotropic solid materials. When glasses are heated to the glass transition (Tg) region, the atomic mobility increases, leading to the formation of crystalline nuclei embedded in an amorphous matrix. Depending on the thermal treatment, these nuclei can grow and give rise to glass-ceramic materials above the crystallisation temperature (Tc). In the GPa region, static pressures can exert an actual catalyst effect on the crystallisation of glass at room temperature [1]. Lithium disilicate glass and glass ceramic have been widely studied at high-pressure, presenting interesting results such as: formation of distinct crystalline phases after different high-pressure and high temperature treatments [2]; and polyamorphism due to the induced densification, resulting in a structure with short range order compatible with the presence of lithium metasilicate [2]. In this work we discuss the in situ analyses by Raman spectroscopy and X-ray diffraction of lithium disilicate (LS2), lithium germanosilicate (LGS), barium disilicate (BS2), and soda lime silica (SLS) glasses and glass-ceramics. These measurements were performed at the EMA beamline in LNLS-Sirius (Brazil) and Xpress beamline in Elettra (Italy) at pressures up to 75 GPa and temperatures up to 623 K. Preliminary results indicate pressure induced crystalline phase transformations on glass ceramic samples, followed by the formation of an amorphous phase, showing a Raman spectrum similar to that shown by pristine glass.
In situ heating under high-pressure also points to phase transitions in BS2 glass ceramic samples. Additionally, ex situ structural and thermal analyses as well as mechanical characterization were performed for retrieved samples, after high-pressure/high temperature processing (up to 7.7 GPa and 1233 K) using the toroidal type chambers available in our laboratory (LAPMA/UFRGS). The ex situ and in situ results are compared and discussed as a function of processing pressure and temperature. Furthermore, a nucleation study was performed using SLS composition, whose results indicate a decrease in the number of nuclei per unit volume for higher pressures.

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Superconductivity in polyhydrides: 8 years after discovery of H3S

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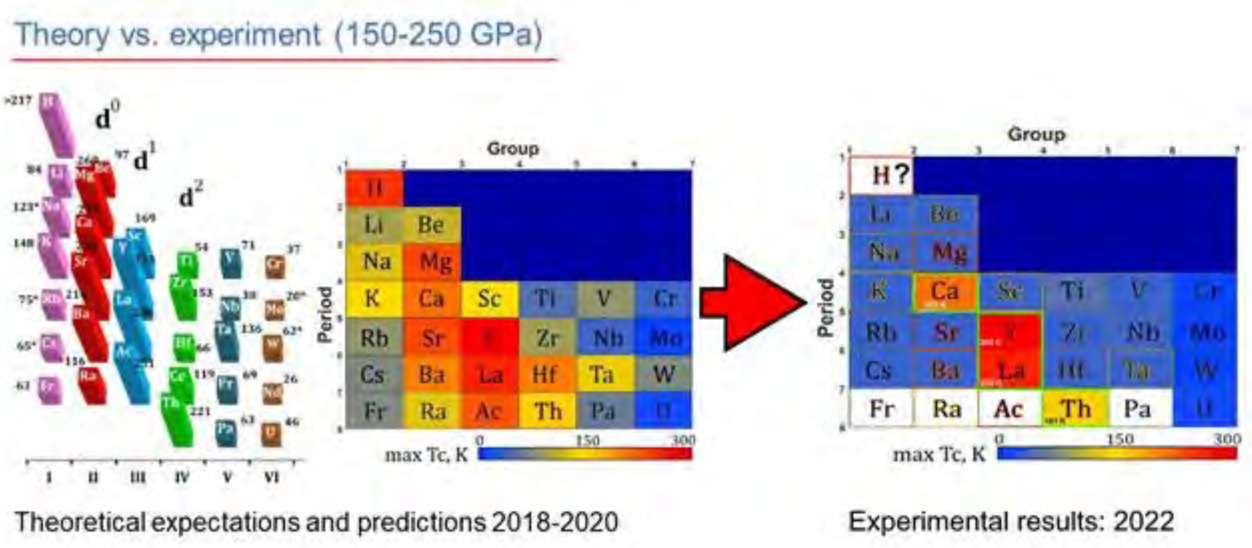
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Over the past 8 years (2015–2023), many polyhydrides have been discovered under pressure. Several groups can be distinguished among them, in particular, molecular polyhydrides, magnetic polyhydrides, and superconducting hydrides ("superhydrides"). The latter have attracted the most attention due to the possibility of reaching room-temperature superconductivity. Moreover, we can already say that a special field has been developed: hydride superconductivity at ultrahigh-pressures. For the most part, the properties of superhydrides are well described by the Migdal–Eliashberg theory of strong electron–phonon interactions, especially when the anharmonicity of phonons is taken into account. I will present a retrospective analysis of data from 2015–2023 and describe promising directions for future research on hydride superconductivity. We will discuss molecular structure of alkali (s^1: Li, Na, K, Rb, Cs) and alkaline earth (s^2: Sr, Ba) polyhydrides. Then I will talk about a small, as it has now become clear, "island of high-Tc superconductivity" among hydrides of d^1-d^2 elements: Ca, Sc, Y, La, Th, Ce, Lu, Zr and Hf. The properties and structure of magnetic polyhydrides of such lanthanides as Nd, Eu, Sm, Gd will also be reviewed. In conclusion, I will focus on the progress in the synthesis and single-crystal X-ray diffraction of tin (SnH_4) and sulfur-carbon $(C,S)H_3$ hydrides, the development of high-pressure NMR, and the investigation of polyhydrides in superstrong magnetic fields.



Theory vs. experiment (150-250 GPa)

Theoretical expectations and predictions 2018-2020

Experimental results: 2022

Superconductivity above 210 K Discovered in Superhydrides of Calcium

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Here, we report that high temperature superconducting calcium polyhydride CaH_6 with H_{24} cage structure was successfully synthesised for the first time. Using metal Ca and ammonia borane, calcium polyhydride samples were successfully synthesised under high temperature and pressure circumstance of 160–205 GPa and 2000 K. The results of in situ high-pressure synchrotron radiation X-ray diffraction experiments indicated that the space group of calcium polyhydride sample is $Im\bar{3}m$, which is the first successful synthesised metal polyhydrides with hydrogen cage structure apart from rare earth metal and actinide metal hydrides. In in-situ high-pressure electrical experiments, the resistance of CaH_6 suddenly decreases and reaches 0 eventually, and the transition temperature gradually decreases with the increase of magnetic field strength, which confirms the superconductivity phenomenon. At 160 GPa, the T_c of CaH_6 is above 210 K, which is the highest T_c among alkaline earth metal hydrides found so far.

Acknowledgement: We would like to thank Professors Haozhe Liu, Luhong Wang, V. B. Prakapenka, S. Chariton, Paul A. Ginsberg, Shuhua Yuan and Jay Bass for collaborations.

[1] Z.W. Li, X. He, C. Q. Jin, et al. Nature Communications 13, 2863(2022)

Superconductivity above 80 K in Polyhydrides of Hafnium

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Studies on polyhydrides are attracting growing attentions recently due to their potential high temperature superconductivity (SC). We here report the discovery of SC in hafnium polyhydrides at high-pressure. The hafnium superhydrides are synthesised at high-pressure and high temperature conditions using diamond anvil cell in combination with in-situ high-pressure laser heating technique. The SC was investigated by in-situ high-pressure resistance measurements in applied magnetic fields. A superconducting transition with onset Tc ~ 83 K was observed at 243 GPa. The upper critical field μ0Hc2(0) was estimated to be 24 T by GL theory and the consequent superconducting coherent length ξ to be ~ 37 Å. Our results suggest that the superconducting phase is from C2/m-HfH14.
This is the first 5d transition metal polyhydride superconductor with Tc above the liquid nitrogen temperature.

Acknowledgement: We would like to thank Professors Haozhe Liu, Luhong Wang, V. B. Prakapenka, and S. Chariton for collaboration.

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Negative linear compressibility in Se under pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Selenium is one of the most classic element materials with rich phase transitions under high-pressure. In this work, a series of in situ synchrotron X-ray diffraction (XRD) measurements was carried out on Se under pressure, with pressure increased from ambient to 210 GPa, negative linear compressibility (NLC) behaviour was first observed from 5.6 GPa to 9.0 GPa with chain structure of Se-I phase, then at pressures between 120 and 148 GPa with β-Po structure of Se-V' phase, the latter of which makes Se the NLC material discovered at the highest pressure so far[1].

Earlier studies by McCann[2] and Keller[3] reported that the NLC behaviour along the Se c-axis occurred immediately from ambient pressure to about 10 GPa. However, in this work, by using Rietveld fitting method on the XRD data, it was discovered that the NLC behaviour along the c-axis only existed from 5.1 GPa to 9.0 GPa, before 5.1 GPa there was a continuous compression along both c-axis and a-axis. The NLC behaviour from 5.1 GPa to 9.0 GPa is attributed to the difference in bonding strengths between interchain and intrachain in the trigonal Se.

With pressure increased to 120 GPa, the NLC behaviour along the a-axis in β-Po structure of Se-V' phase was discovered. The appearance of NLC behaviour makes the bulk modulus of Se from 120 to 148 GPa the highest (321 ± 2 GPa) compared to the bulk modulus at pressures lower than 120 GPa (83 ± 2 GPa) or higher than 148 GPa (266 ±7 GPa). The discovery of NLC material in a higher-pressure range is always more significant in terms of fundamental mechanism and new materials understanding, yet it has barely been reported at pressures above 100 GPa. The current discovery of NLC in Se will hopefully inspire future studies on other potential NLC materials at ultra-high pressure.

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In situ X-ray diffraction study of (Fe, Mg)O under shock compression

Khachiwan Buakor[1], M. Harmand[2], J. Pintor[2], E. Cunningham[3], T. Duffy[4], A. Gleason[3], D. Khaghani[3], D. Kim[4], Z. Konopkova[1], Hae Ja Lee[3], I. Ocampo[4], C. Plueckthun[5], Ronald Redmer[6], R. Smith[7], M. Schoelmerich[7], S. Speziale[8], S. Tracy[9], H. E. Tsai[3], T. Tschentscher[1], K. Appel[1]

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Over 5000 exoplanets including super-Earths, a class of rocky planets with 2 to 10 Earth masses, have been discovered in the past decades. Understanding and modeling the structure and formation of super-Earths strongly rely on the knowledge of phase stability and physical properties at extreme conditions of relevant compounds. (Fe,Mg)O is considered to be one of the major phases of the Earth’s and Super Earth’s mantle. It has been the subject of intensive investigation including structural and electronic phase transition. However, the properties of (Fe,Mg)O solid solutions at ultra-high pressure, i.e. the pressure relevant to planetary interiors, are still mostly unknown.

Here we present our X-ray diffraction (XRD) study of several compositions in the (Fe,Mg)O solid solution series under laser-driven shock compression. We aim to investigate phase transitions and the solidus-liquidus boundaries of (Fe,Mg)O along their Hugoniot to megabar pressure. (Fe,Mg)O targets were prepared with different Fe contents ranging from 10 to 80 percent to determine the effects of Fe-Mg substitution on (Fe,Mg)O phase behaviours. The XRD measurements of compressed (Fe,Mg)O were made using femtosecond XFEL pulses and performing time scans with nanosecond steps around the shock breakout. The preliminary results show that we observed the solid B1-phase of (Fe,Mg)O for all the compositions up to the maximum pressure at around 140 GPa and possibly observed the melting of Fe-rich (Fe,Mg)O.

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Green luminescence in heavily carbon doped GaN synthesised by atomic substitution under high-pressure and high temperature

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Gallium nitride is a wide-band-gap semiconductor with many important applications such as short-wavelength, light-emitting diodes, high-frequency transistors and power electronic devices [1,2]. Carbon doping in gallium nitride has become an important research topic due to its potential in realizing semi-insulating gallium nitride, in addition to its role in the continuous search for high-efficiency P-type doping [3]. As well known, a small amount of carbon doping can cause the yellow luminescence in GaN [4-6]. In this work, the luminescence of heavily carbon doped GaN obtained by atomic substitution under high-pressure and high temperature was investigated. The single crystal of C doped GaN was synthesised from GaN single crystal and excessive carbon under 20GPa and 2273K. Photoluminescence spectra (PL spectra) obtained at 300 K shows the green band at ~522 nm as shown in figure 1. This phenomenon provides a possible way to adjust the yellow-green fluorescence of GaN by controlling the amount of carbon doping, and then realise the continuous adjustment of GaN band gap.

Intermolecular interactions in multicomponent crystals under pressure: a study case of 1,2-bis(4-pyridyl)ethane and fumaric acid cocrystal

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Multicomponent crystals are an extremely important group of materials for crystal engineering. [1] Their diversification and modularity allows forging the chemical environment that cannot be formed in crystals of pure compounds. This translates into unique networks of intermolecular interactions and, as a consequence, design of novel materials of desired physicochemical properties. [2] The potential of multicomponent crystals is visible in the Crystal Structure Database (CSD), [3] among over 1.2 million deposits, 44.51% represent multicomponent crystals.

On the other hand, research aiming to enhance potential of multicomponent crystals by using high-pressure, despite captivating results, [4-6] are still uncommon. In fact, only 10.83% of high-pressure deposits contain at least two different chemical entities (cocrystals: 28.11%, salts: 34.56%, hydrates: 28.11% and other solvates: 10.83%).

Herein, we present results of the high-pressure study of a model cocrystal of 1,2-bis(4-pyridyl)ethane and fumaric acid in the context of statistical analysis of the CSD, focused on changes in the strong and weak hydrogen bonds, such as O-H···N and C-H···N bonds.[7] The single crystal of 1,2-bis(4-pyridyl)ethane and fumaric acid (ETYFUM) cocrystal was compressed isothermally in Merrill-Bassett [8] diamond anvil cell up to ca 3 GPa with Daphne 7575 oil used as hydrostatic medium. At each pressure step, the X-ray diffraction experiment was performed using 4-circle X-ray diffractometer equipped in Mo Kα X-ray tube and EOS CCDC detector. The statistical analysis was performed using CSD API Python on CSD database, version 5.43 (November 2022). [9]

The transformation of the crystal of ETYFUM on the compression to 3 GPa is discussed. The molecular packing and intermolecular interactions are analysed with focus on the interactions between the coformers. The results are put into perspective of the changes observed for similar interactions occurring in previously reported structures deposited in the CSD.

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In situ study of Fe2O3 at pressure and temperature conditions of the Earth's lower mantle

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Iron is the most abundant transition metal in the Earth's interior. It occurs predominantly as ferric and ferrous iron and exhibits a complex chemistry in compounds at high-pressure (p) and temperature (T). Knowledge about the electronic structure of iron-bearing compounds, e.g. spin- and oxidation state as well as coordination, is of particular relevance for a better understanding of deep Earth processes. In this context, the p/T-induced structural and electronic transitions of Fe_2O_3 are of high interest to elucidate the role of ferric iron regarding chemistry, redox conditions and dynamics of the Earth's mantle. However, the electronic structure of its different high-pressure phases is widely unexplored at high temperature and therefore a subject of recent research [1–10].

We performed (resonant) X-ray emission spectroscopy ((R)XES) to study iron in Fe_2O_3 at beamline P01 at PETRAIII (DESY) utilizing a wavelength dispersive von Hámos spectrometer [11,12]. We used diamond anvil cells in combination with a double-sided Yb:YAG-laser heating setup [13] to achieve pressure and temperature conditions up to 80 GPa and 2500 K, respectively.

For the measurements at ambient temperature, we focused on the alpha-, zeta- and theta-phases with iron in the high, mixed and low spin states. In the phase with mixed spin state, the iron is separated into two distinct coordinations in high spin and low spin state, respectively. Starting from these phases, the samples were laser heated to p/T conditions of the phase stability regime of the iota- and eta-phase, probing iron's electronic state in situ via XES in the $K\beta_{1\,3}$ and valence-to-core (vtc) regime and via RXES at ambient temperature using the Kα emission before heating and after temperature quenching.

For the mixed spin phases at ambient temperature the spin state results indicate a distortion on the HS spin site. At pressures of 64 and 80 GPa, we observe an increase in spin during heating compared to the initial value, as well as a partial relaxation of the spin state after temperature quenching. Further we will present our finding on the vtc development depending on the temperature as well as the RXES analysis on the electronic structure of the iron before and after laser heating.

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Elastic moduli, anisotropy and refractive index of γ-Ge_3N_4 via laser ultrasonics, Brillouin light scattering, and first-principles calculations

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Germanium nitride having cubic spinel structure, γ-Ge_3N_4, is a wide band-gap semiconductor, Eg=3.65(5) eV, with a large exciton binding energy [1,2] which exhibits high hardness, elastic moduli and elevated thermal stability to ~700°C. Experimental data on its bulk and shear moduli (B_0 and G_0, respectively) are strongly limited [3–6], inconsistent and, thus, require verification. Moreover, earlier first-principles density functional calculations provided significantly scattering B_0 values but consistently predicted G_0 much higher than the experimental one [7–9]. Here, we investigated the elastic behaviour of γ-Ge_3N_4 experimentally, using the techniques of laser ultrasonics (LU) and Brillouin light scattering (BLS), and theoretically, using the first-principles calculations. We measured velocities of the Rayleigh surface acoustic waves (RSAWs) and of the longitudinal acoustic waves (LAWs) in our low-porosity polycrystalline sample using the LU-technique and derived its isotropic elastic moduli Bp and Gp (here and below, the subscripts p and 0 indicate values for the porous and densified sample, respectively). Applying the Hashin-Shtrikman approach, we obtained B_0=322(44) GPa and G_0=188(7) GPa for the dense γ-Ge_3N_4. The significant error in B_0 is explained by the low amplitude of the LAW-signals in our LU-measurements. Nevertheless, it agrees, within experimental uncertainties, with the B_0=295(5) GPa [3] obtained from the EOS measured upon quasi-hydrostatic compression. Both the previous and the present first-principles calculations underestimated B_0 while the GGA-PBEsol functional with a hard PAW potential showed the least deviation from the experiment.

Our experimental G_0 exceeds by ~1.5 times the previous experimental value, G_0=124-129 GPa derived from the nanoindentation testing [4, 5]. The calculated G_0 values, typically around 170 GPa, were underestimated but much less significant than the B_0, between 211-269 GPa. However, G_0=186(10) GPa calculated using the GGA-PBEsol functional with a hard PAW potential agreed with our measurements. Comparison of the RSAW-velocity values, VR_0, measured using the LU- and BLS techniques confirmed the theoretically-predicted elastic anisotropy of γ-Ge_3N_4. The anisotropy led to the asymmetric RSAW peaks in our BLS measurements resulting in the VR_0 underestimation by ~10%. Combining our LU- and BLS results for the LAWs, we determined the refractive index of the dense γ-Ge_3N_4 to be n_0=2.4(1). This value ranks γ-Ge_3N_4 in the class of very-high-refractive-index materials transparent for visible light, such as diamond, GaN or TiO_2 having anatase structure.

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Studies of PFOA at extreme conditions

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Perfluoroalkyl substances (PFAS) are a class of fluorine-bearing compounds known as "forever chemicals" due to their high chemical stability and resistance to chemical breakdown. Due to various industrial chemical processes that use fluorine plastics, they have been found in increasing numbers worldwide. As these chemicals are toxic and are carcinogens, there is much interest in finding means to break down these chemicals as they build up in our environment.

We sought to investigate whether or not a combination of modest high-pressure and X-ray irradiation would break down these challenging substances. To start, we chose to examine the behaviour of perfluorooctanoic acid (PFOA) up to 25 GPa using Raman, far- and mid-IR spectroscopy. We observed two phase transitions at high-pressure at 2 GPa and 8 GPa, respectively.

We also performed a separate synchrotron white X-ray ray irradiation study of PFOA at 2 GPa and observed dramatic and irreversible transformation of the compound after 1 hour of irradiation where the solid compound turned into a liquid and disappearance of the Raman spectrum when decompressed to ambient pressure. This result suggests that a combination of modest pressure and irradiation may be able to easily break down PFOA into simpler constituents that may be more easily isolated and/or chemically treated/denatured.

To the best of our knowledge, this is the first study of PFOA at high-pressure.

High-Pressure Effect on EuNi2As2

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Since the discovery of iron-based superconductors, the ternary 122-phase with the ThCr2Si2 type structure has been studied well due to its high superconducting transition temperature up to 38 K. EuFe2As2 exhibits superconductivity up to 32 K by chemical doping or external pressure application. It is interesting to know whether the isostructural EuNi2As2 exhibits superconductivity by modification similar to the Fe system. In this study, we prepared single crystals of EuNi2As2 and investigated the pressure effect on them.

Crystal-field mediated electronic transitions of EuX monochalcogenides (X = O, S, Se and Te) up to 35 GPs

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Eu^{2}+ has always attracted scientists of different fields in Chemistry, Physics and Materials Science and Technology due to its unique electronic structure characterised by its $4f^7 5d^0$ ground and $4f^6 5d^1$ excited state. The Eu^{2}+ is one of the most investigated activator in highly efficient phosphors. Furthermore, the outer 5d electrons strongly interact with the surrounding ions (ligands) allowing the tunability of the photoluminescence wavelength from red to blue just by selecting the appropriate host site structure. EuX monochalcogenides (X: O, S, Se, Te) offer an excellent opportunity to study the pressure dependence of the correlation between the electronic and the geometric structures in Eu^{2}+, especially in view of the simple cubic structures and regular local environments. EuX crystallises in the NaCl-type structure (B1, space group Fm-3m), and undergoes a phase transition to the high-pressure CsCl-type structure (B2, space group Pm-3m) under compression. Thus, Eu^{2}+ experiences an increase of the coordination from octahedral (EuX_6) to hexahedral (EuX_8). The transition pressure is higher as the ionic radius of X decreases. The EuO, EuS, EuSe and EuTe series undergoes the structural phase transition at 50, 21.5, 18 and 10.5 GPa, respectively.

Here, we present a detailed experimental and theoretical analysis of the pressure dependence of the electronic structure of EuS up to 35 GPa. The electronic properties have been studied by optical absorption in the laboratory of Universidad de Cantabria and by XANES spectroscopy (X-ray absorption near edge structure) at BM23 beamline in the European Synchrotron Radiation Facility (ESRF). Ab initio calculations within the density functional theory (DFT) formalism, by using VASP (Vienna Ab initio Simulation Package), have been carried out to explain the experimental evidence. We show two electronic transitions from semiconductor to semi-metal at 14 GPa and semi-metal to metal at 21.5 GPa. The responsible strength of these electronic modifications is the crystal field at the Eu^{2}+ site in the structure of EuS. Likewise, this conclusion can be extended to the whole EuX series.

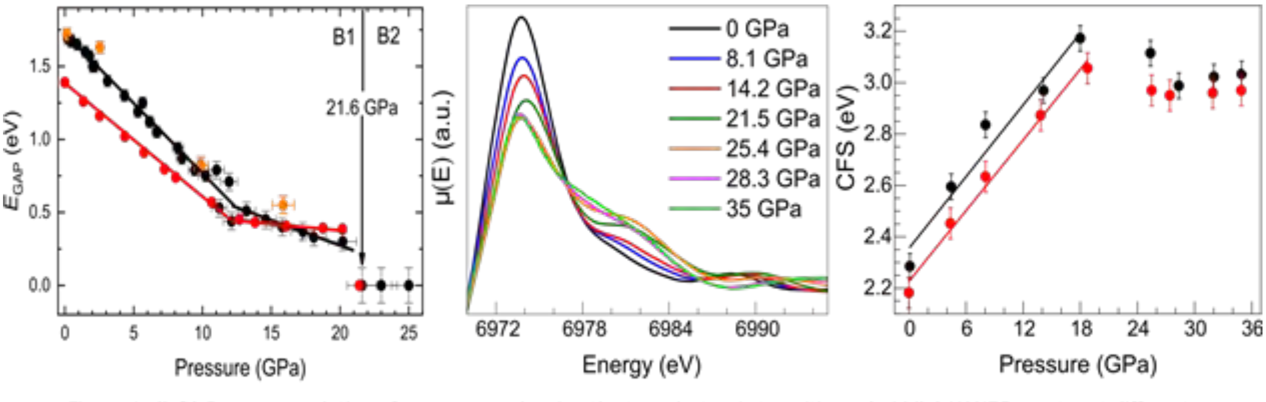

Figure 1. (left) Pressure evolution of energy gap showing the two electronic transitions, (middle) XANES spectra at different pressures, and (right) crystal-field splitting under pressure up to 35 GPa.

A new metastable state in the rare-earth hexaboride EuB_6 induced by high-pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Both technology and fundamental science drive intense experimental and theoretical research on a variety of metal borides [1–4]. The transition metal borides are known for their high shear strength and have been studied to ultra-high pressures [5], however, similar studies on rare-earth borides are limited. In the series of the rare-earth hexaborides, EuB_6 shows unique electrical transport, magnetic, and topological properties. In addition, it is the only hexaboride with ferromagnetic order and a low thermal expansion coefficient, which can be tuned to the ground state electron system [6,7]. Its simple cubic (space group: Pm-3m, no. 221), but relatively robust structure, demonstrates its intrinsic hard nature [7]. Here, Eu atoms are located at the corners of the cubic lattice and six B atoms form an octahedral cage inside the cube. The enhanced mechanical hardness likely stems from the increase in valence electron density from europium, which contributes to the strong covalent network. The incompressibility and stability of the caged EuB_6 system under extreme conditions are of interest, especially after nanoscale amorphous shear bands were observed by using a nanoindentation approach [6]. To study this further, we performed synchrotron powder angular-dispersive X-ray diffraction (ADXRD) measurements using the diamond anvil cell technique for pressures up to 180 GPa. Our study shows that the structure of EuB_6 is stable up to 82 GPa, whereupon a sudden and highly diffuse expansion of the rings in the ADXRD data is observed. This indicates that the periodic arrangement of atoms in the EuB_6 crystal has been disrupted, causing the formation of a new, amorphous-disordered-type phase that lacks the long-range order characteristic of crystalline solids. The metastable disordered phase of EuB_6 is retained on decompression from |180 GPa. Such a phase transition from a cubic phase is quite unique: to the best of our knowledge, EuB_6 is the first rare-earth hexaboride to deviate from the well-defined ordered crystal structures.

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Magnetism of geometrically frustrated Yb2Pt2Pb in elevated pressures

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Yb2Pt2Pb crystallizing in a tetragonal U2Pt2Sn-type structure represents an example of metallic Shastry-Sutherland system allowing to examine the interplay between geometrical frustration (arising from the dimerization of Yb ions in basal planes) and f-conduction electron hybridization. Since both Kondo screening and frustration play important role in Yb2Pt2Pb physics, this system potentially allows experimental exploration of universal phase diagram suggested for heavy-fermion compounds [1,2]. Yb2Pt2Pb is the TN ≈ 2 K antiferromagnet with magnetic order arising from strongly fluctuating paramagnetic liquid of Yb dimers. Complex magnetic phase diagrams were reported in dependence on the direction of applied magnetic field and several magnetization steps were observed as the typical feature of geometrically frustrated magnetism [2,3].

Herewith, we have focused on the pressure impact on the magnetism of Yb2Pt2Pb. Application of mechanical pressure is the pure technique allowing to act directly on the crystal lattice without impact on the sample purity. Changes of the nearest and the second nearest Yb distances may affect the relation of intradimer and interdimer interactions standing behind the Shastry-Sutherland frustration. Since the magnetic properties of Yb2Pt2Pb are strongly anisotropic, the single-crystalline samples were prepared by Pb-self flux growth technique. The first experiments in elevated pressures have shown surprisingly high stability of magnetic order with almost no change in the Néel temperature in pressures up to 2 GPa. On the other hand, the pressure impact on the fields at which metamagnetic transitions are observed is quite strong. In case of magnetic field applied along the crystallographic c-direction, the metamagnetic transition field changes with more than 3 T/GPa. The experimental range of magnetic field applied in the c-direction was extended with respect to the previously published data. This allows to enrich the phase diagram with the fields above the metamagnetic transition where the suppression of Néel temperature starts to be more significant in contrast to its high stability in moderate magnetic fields.

Recently gained results of both pressure and magnetic field response of the studied system as well as the crystal anisotropy aspects will be presented in the conference contribution.

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Investigating the melting temperature of silicates at extreme conditions using single pulse laser heating and time-resolved XRD

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The high energy density instrument (HED) at the European X-ray free-electron laser (EuXFEL) provides unique new capabilities for experimental investigation of matter at extreme conditions. The huge brightness enables single pulse X-ray diffraction images with up to 4.5 MHz repetition rate. Thus, enabling the unprecedented opportunity to study time-resolved response of materials to e.g. <1us single laser pulses under high-pressure.

Using a combination of single pulse laser heating, X-ray Diffraction and diamond anvil cells, we investigated the melting behaviour of Fe-bearing silicate glasses at high-pressures. By using this method, we were able to observe material responses with high time resolution and with much less interference of chemical reactions from the surrounding environment compared to previous attempts, due to the short duration of the experiment (~20us). The data was collected at the HED, during two community proposals which brought together around fifty international participants (proposal numbers #2292 and #2605).

Following the preliminary results of the experiment, we aim to identify the pressure and temperature conditions under which the silicate melts crystallise by analyzing the emergence of new XRD diffraction peaks upon cooling from up to 10.000 K, whereby the temperature evolution is estimated using streak optical pyrometry (SOP).

We will present first results and challenges for this kind of experiment and give an outlook for future investigations of melting temperatures for similar materials using this unique new approach.

Raman study of supercritical fluid hydrogen coexisting with multilayer graphene at room temperature

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Phase diagram of hydrogen, in particular solid phase was investigated by many researchers around the world. However, we don't know the physical properties of super-critical fluid-phase of hydrogen very much. To utilise hydrogen easily, effectively, and safely as the future clean energy, we should know the physical properties of fluid hydrogen. Furthermore, one of the important things to understand about the properties of hydrogen is clarify the effects of hydrogen on host materials. In our previous study, nano-carbon materials with various framework, for example, multiwalled carbon nanotubes, were compressed with hydrogen gas in a diamond anvil cell (DAC) at room temperature [1]. At the pressures of 0.56 GPa, we had a clue that carbon-carbon bonding composing the framework causes unusual changes because of hydrogen, but we could not know the mechanism. Recently a fluid-fluid phase-transition of hydrogen was revealed at 0.56 GPa in the supercritical-fluid phase at room temperature [2]. The anomalous changes in the carbon framework we observed were suggested to be due to an intrinsic hydrogen transition. To investigate the effect of fluid hydrogen on the carbon framework, multilayer graphene having a simple structure was chosen as the host material in this study. Multilayer graphene whose number of layers are countable and Raman spectroscopy with confocal optics, can capture the motion of hydrogen molecule incorporated between the layers. DAC was used for loading the pressure. Multilayer graphene with different number of layers were transferred to the culet surface of lower diamond anvil. The number of layers were estimated from the Raman-spectral intensity-ratios of the substrate diamonds [3]. Ruby balls for the pressure marker were placed on the culet of the upper anvil. Silver-cooper alloy having hydrogen resistance was used as a gasket material. The sample was prepared by filling the gasket hole with high-density hydrogen-gas composed up to 180 MPa at room temperature using a gas loading system at NIMS. A 180-degree backscattering-geometry was carried out for the Raman-spectral measurements using an excitation laser light with a wavelength of 532.28 nm. Details on how to focus on interlayer hydrogen for Raman measurements will be explained in our presentation.

We obtained rotational and vibrational spectra of hydrogen were obtained in addition to the G-band from multilayer graphene by pressurization up to 1.5 GPa. Anomalous pressure changes in the Raman shifts, which may correspond to a gas-fluid transition at pressure above 0.2 GPa, were observed in the rotational and vibrational bands. They also showed a jump in the pressure change at around 0.42 GPa, which is lower than the fluid-fluid transition pressure observed in pure hydrogen [3].
In addition, the change in the Raman intensity ratio of rotational spectra for J=0 and 1 appeared above 0.8 GPa (J is the angular-rotational quantum-number), and the ratio of para-hydrogen showed maximum around 1.2 GPa.

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Crystal structure and superconductivity of alkaline earth metal Strontium at low temperature and high-pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The high-pressure phases of the alkaline-earth metals were investigated experimentally and theoretically. The first principle calculations for these elements show an increase of the d-band occupation with increasing pressure [1]. Structural experiments have revealed that a “host-guest” structure is formed in the high-pressure of Ca, Sr and Ba [2,3,4]. Both superconducting transition temperatures (Tc) have a maximum point in the host-guest phase stable region of Sr and Ba, where the s-d electron transfer is considered to be completed. The host-guest structure with modulating is proposed to suppress the enhancement of Tc, which is attributed to the formation of charge density wave [5,6].

Recently, a new phase of Ba (Ba-VI) was discovered by the compression at low temperature [7]. And Tc increased with pressure in Ba-VI and about 8K of maximum Tc was recorded [8].

Strontium transforms from the ambient-pressure face-centred cubic phase, to a body-centred cubic structure at 3.5 GPa. This phase is stable up to 24 GPa, where there is a transition to Sr-III. On further compression, there are transitions to Sr-IV at 35 GPa, and to Sr-V at 46 GPa. Dunn and Bundy reported the discovery of pressure-induced superconductivity in Sr at pressures from 35 to 50 GPa [9]. Tc rises with increasing pressure and reaches 8 K and 58 GPa. In order to understand systematically the structural phase transition and superconductivity in alkaline-earth metals at high-pressure, we investigated X-ray diffraction measurement and electrical resistance measurement of Sr under high-pressure and low temperature.

The pressure was applied at room temperature and then the diamond anvil cell was cooled down in a cryostat. The X-ray diffraction experiments were carried out by an angle-dispersive method using an image-plate detector. X-ray diffraction patterns of Sr at high-pressure and low-temperature were obtained by the synchrotron radiation at BL10XU in SPring-8. In powder X-ray diffraction at 10 K, Sr with increasing pressure transitions from bcc to Sr-III and to Sr-IV phases at almost same pressure to that at room temperature. By further compression, new spectrums appeared indicating transitions to new high-pressure phases (Sr-VI, Sr-VII). And Tc rose to 14 K at 150 GPa in Sr-VI with increasing pressure, which was higher than the maximum Tc in host-guest structure phase (Sr-V).

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Machine Learning Accelerated Simulation of Solid-Solid Phase Transitions under High Pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Simulating reconstructive phase transition in solid materials requires an accurate description of potential energy surface (PES). Density-functional-theory (DFT) based molecular dynamics can achieve the desired accuracy, but it is computationally unfeasible for large systems and/or long simulation times. In this talk, I will introduce a new approach for simulating reconstructive phase transitions, which combines metadynamics simulation and machine learning representation of high-dimensional PES. The method is resulted from the collaborations with researchers at Jilin University and University of Nevada. The new method can reach the accuracy on par with the simulations based on DFT, but with the computational cost several orders of magnitude less, and scaling with system size approximately linear. The high accuracy of the method will be demonstrated in the simulation of pressure-induced phase transitions in gallium nitride (GaN). The large-scale simulation using a half-million atom simulation box reveals the phase transition with excellent detail, revealing a sequential change of phase transition mechanism from collective modes to nucleation and growths. The observed change of atomistic mechanism manifests the importance of statistical sampling with large system size. The combination of MLP and metadynamics is likely to be applicable to a broad class of induced reconstructive phase transitions at extreme conditions.

Pressure-induced magnetic transition of ε-FeOOH at 8 GPa

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

ε-FeOOH is a high-pressure polymorph of goethite (α-FeOOH) and has been reported to play a role in water transportation by subducting slabs at the mantle transition zone and the lower mantle. It has the InOOH-type orthorhombic structure with a single octahedral site occupied by ferric iron. At high-pressure, it has been reported to have two transitions without major structural modification: hydrogen bond symmetrisation at ~18 GPa (between asymmetric and disordered states) and electronic spin transition at 45 GPa. In this report, we will present one more transition, which is a magnetic spin-flop at 8 GPa (Ikeda et al., 2023).

ε-FeOOH is antiferromagnetic at ambient pressure and temperature (Pernet et al., 1973; 1975), and its magnetic properties at high-pressure have not been reported. Here, we conducted Mössbauer spectroscopic measurements of polycrystalline ε-^{57}FeOOH at high-pressure. The sample was synthesised in advance from a mixture of $^{57}Fe_2O_3$ powder and distilled water at 8 GPa and 700 K. Symmetric diamond anvil cells (DACs) with 4:1 Methanol–ethanol pressure medium were used to produce high-pressure for the Mössbauer measurements. Pressure conditions were estimated using the ruby-fluorescence method. Energy domain ^{57}Fe-Mössbauer measurements were conducted using a nuclear Bragg monochromator at the BL11XU beamline of SPring-8, Japan (Mitsui et al., 2009). The obtained spectra were fitted using the MossA program (Prescher et al., 2012).

Mössbauer spectra of polycrystalline ε-FeOOH were obtained from 0 to 14.6 GPa tracking compression and decompression in identical DACs. A single sextet model with quadrupole splitting provided a good fit for every spectrum. During compression, the quadrupole shift 2ε changed from negative to positive at around 8 GPa, implying a change in the direction of the magnetic moment. The spin-rotation angle is calculated from the 2ε transition to be in the range 0 to 44.8(2)°, using the assumptions $e^2qQ \ll \mu H$, an axially symmetric electric field, and constant e^2qQ in the transition pressure range. The transition has not been reported despite experimental studies on ε-FeOOH, including powder X-ray diffraction experiments and sound velocity measurements. Therefore, the transition is apparently not accompanied by a structural change and would have minimal effect on elastic properties.

Hydrostatic behaviour of selected chemically inert pressure-transmitting media

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The pressure-transmitting medium (PTM) is crucial for many high-pressure experiments, therefore, the hydrostatic properties of various substances have been studied [1,2]. It has been shown that solidified gases such as H2, He, and Ne have the highest hydrostatic limit [1]. However, special gas loading devices are required for their use and such instrumentation is often limited to special facilities. For high-pressure single-crystal X-ray diffraction experiments [3] in the pressure range up to 10 GPa, commonly used pressure-transmitting media are ambient-condition liquids, such as, methanol–ethanol mixture (4:1), isopentane–n-pentane mixture (1:1), Daphne oils and silicone oils [2–4]. The choice of PTM depends on the targeted experimental pressure range, sample solubility, and the possible reactivity of the sample with the pressurised medium. The selection of PTM for reactive samples, such as the noble-gas compounds, which are powerful oxidising and fluorinating agents, is essentially limited to perfluorinated liquids. In this case, the only thoroughly studied PTM are Fluorinert liquids [4–6], which are hydrostatic only up to about 2 GPa. Another class of inert perfluorinated liquids are perfluoropolyethers or Fomblin oils, where only the highly viscous Fomblin Y 140/13 has been investigated as potential PTM and shown to be useful only up to 1 GPa [7].

In this work, selected inert fluorinated liquids were investigated as potential pressure-transmitting media. These liquids have negligible vapour pressure, are easy to handle, and do not dissolve a vast majority of compounds. In our experiments, the investigated media have proven to be resistive towards oxidative fluorination by XeF2. To gain insight into their hydrostatic properties, the liquids were loaded into Merrill-Bassett diamond-anvil cells and several ruby spheres were placed in the pressure chambers. The hydrostatic behaviour of the studied pressure medium was inferred from the pressure distribution across the sample chamber [2,4], which was probed by fluorescence measurements on each ruby at each pressure increase step.

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Simulations of multivariant Si I to Si II phase transformation in polycrystalline silicon with finite-strain scale-free phase-field approach

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Scale-free phase-field approach (PFA) at large strains and corresponding finite element method (FEM) [1] simulations for multivariant martensitic phase transformation (PT) from cubic Si I to tetragonal Si II in a polycrystalline aggregate are presented [2]. Important features of the model are a large and very anisotropic transformation strain tensor εt = {0.1753; 0.1753; −0.447} and stress-tensor dependent athermal dissipative threshold [3,4] for the PT, which produces essential challenges for computational treatment. 3D polycrystals with 55 and 910 stochastically oriented grains have been subjected to uniaxial strain- and stress-controlled loadings under periodic boundary conditions and zero averaged lateral strains. Coupled evolution of discrete martensitic microstructure, volume fractions of martensitic variants and Si II, stress and transformation strain tensors, and texture are presented and analysed. Macroscopic variables effectively representing multivariant transformational behaviour are introduced. Macroscopic (overall) stress-strain and transformational behaviour for 55 and 910 grains are quite close and differ by less than 10%. This allows the determination of the macroscopic constitutive equations by treating aggregate with a small number of grains. Large transformation strains and grain boundaries lead to huge internal stresses of tens GPa, which affect microstructure evolution and macroscopic behaviour. In contrast to a single crystal, the local mechanical instabilities due to PT and negative local tangent modulus are stabilised at the macroscale by arresting/slowing the growth of Si II regions by the grain boundaries and generating the internal back stresses. This leads to increasing stress during PT. The developed methodology can be used for studying various PTs with large transformation strains (e.g., graphite to diamond, graphite-like BN to superhard diamond-like BN, PTs in Ge and GaSb, etc.) and for further development by including plastic strain and strain-induced PTs.

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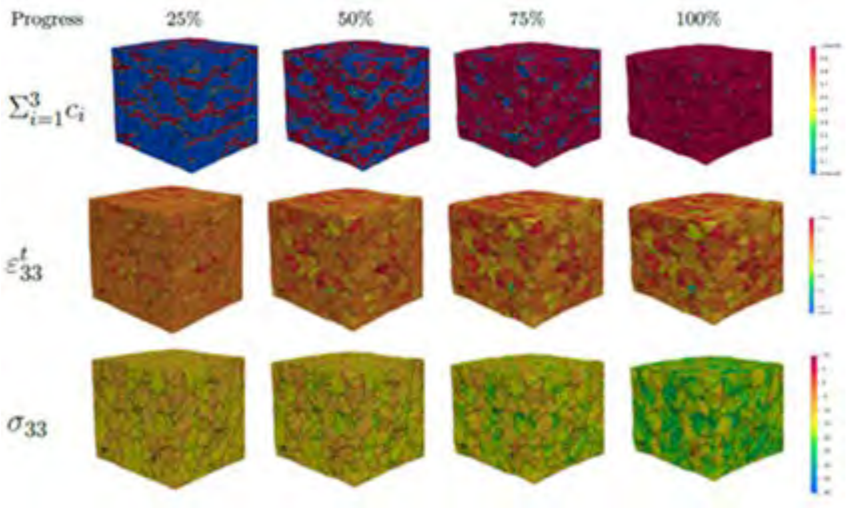
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Plastic strain-induced phase transformations in Si: drastic reduction of transformation pressures, change in transformation sequence, and size effect

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Pressure-induced phase transformations (PTs) between numerous phases of Si, the most important electronic material, have been studied for decades. This is not the case for plastic strain-induced PTs. The pressure-induced PTs are initiated by nucleation at the pre-existing defects, like dislocations or grain boundaries, whereas the plastic strain-induced PTs occur at new defects constantly generated during the plastic flow [1.2]. In this study, we revealed in-situ various unexpected plastic strain-induced PT phenomena. The hydrostatic, plastic compression, and shear-induced PT studies were conducted on Si with particle sizes of d = 1 µm, 100 nm, and 30 nm. X-ray diffraction experiments were carried out at the 16-ID-B beam line, HPCAT utilizing Advanced Photon Source with X-ray wavelengths 0.4133 Å and 0.3445 Å. The XRD patterns were refined using GSAS-II software to extract the lattice parameters and volume fractions of Si phases using the Rietveld refinement method.

We predicted theoretically (opposite to the current theory) that when a reduction in the grain size leads to an increase in the yield strength (Hall-Petch effect) and then to the reduction in the yield strength (inverse Hall-Petch effect), the minimum pressure for strain-induced PT first reduces and then increases. This was confirmed for the appearance of Si-II and is opposite to what we observed in the hydrostatic experiments. For Si powder with an average particle size of 100 nm, strain-induced PT Si-I→Si-II initiates at 0.4 GPa versus 16.2 GPa under hydrostatic conditions (i.e., pressure is reduced by a factor of 40.2!); for 30 nm Si, it is 6.1 GPa versus ∞, since this PT does not occur at all. Similarly, strain-induced PT Si-I→Si-III initiates at 0.6 GPa, while it does not occur under hydrostatic loading. For Si-I→Si-II, the minimum pressure for initiation of strain-induced PT in 100 nm Si, 0.4 GPa, is the same for compression in DAC and torsion in RDAC, i.e., it is independent of the plastic strain tensor and its path, which has multiple practical consequences. Also, plastic straining changes the sequence of PTs and leads to the coexistence of four Si phases. For 100 nm Si, an intermediate Si-III phase between Si-II and Si-XI is observed for the first time under plastic shear. Based on gained understanding, we resolved long-standing puzzles, namely, to retain Si-II at ambient pressure and obtain reverse Si-II→Si-I PT, demonstrating the possibilities of manipulating different synthetic paths [3]. Obtained results have numerous applications for developing economic defect-induced material synthesis, surface treatment (polishing, turning, etc.), and friction.

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Hydrostatic Compression

Micron Si: $\mathrm{I} \xrightarrow{13.5\text{ GPa}} \mathrm{I+II+XI} \xrightarrow{15.3\text{ GPa}} \mathrm{XI} \xrightarrow{16.5\text{ GPa}} \mathrm{V} \xrightarrow{0\text{ GPa}} \mathrm{XII+III}$ (PTM: He)
100 nm Si: $\mathrm{I} \xrightarrow{16.2\text{ GPa}} \mathrm{I+II+XI} \xrightarrow{18.1\text{ GPa}} \mathrm{XI} \xrightarrow{19.3\text{ GPa}} \mathrm{V} \xrightarrow{0\text{ GPa}} \mathrm{XII+III}$ (PTM: He)
30nm Si: $\mathrm{I} \xrightarrow{14.6\text{ GPa}} \mathrm{I+XI} \xrightarrow{19.8\text{ GPa}} \mathrm{I+XI+V} \xrightarrow{21.6\text{ GPa}} \mathrm{I+V} \xrightarrow{23.2\text{ GPa}} \mathrm{V} \xrightarrow{0\text{ GPa}} \mathrm{a}$ (PTM:He)

Non-hydrostatic Compression

Micron Si: $\mathrm{I} \xrightarrow{2.5\text{ GPa}} \mathrm{I+II} \xrightarrow{8.8\text{ GPa}} \mathrm{I+II+XI} \xrightarrow{14.1\text{ GPa}} \mathrm{XI} \xrightarrow{14.2\text{ GPa}} \mathrm{XI+V} \xrightarrow{14.8\text{ GPa}} \mathrm{V} \xrightarrow{0\text{ GPa}} \mathrm{XII+III}$ (S.S.)
Micron Si: $\mathrm{I} \xrightarrow{2.2\text{ GPa}} \mathrm{II}$ (Cu)
100 nm Si: $\mathrm{I} \xrightarrow{0.4\text{ GPa}} \mathrm{I+II} \xrightarrow{13\text{ GPa}} \mathrm{I+II+XI} \xrightarrow{14.4\text{ GPa}} \mathrm{II+XI} \xrightarrow{17.2\text{ GPa}} \mathrm{XI+V} \xrightarrow{18.2\text{ GPa}} \mathrm{V} \xrightarrow{0\text{ GPa}} \mathrm{III}$ (Cu)
30nm Si: $\mathrm{I} \xrightarrow{6.1\text{ GPa}} \mathrm{I+II} \xrightarrow{9.9\text{ GPa}} \mathrm{I+II+XI} \xrightarrow{11.8\text{ GPa}} \mathrm{I+II+XI+V} \xrightarrow{12.6\text{ GPa}} \mathrm{I+XI+V}$ (Cu)
$\mathrm{II\ \&I+II} \xleftarrow{0\text{ GPa}} \mathrm{V} \xleftarrow{13.4\text{ GPa}}$

Torsion

100 nm Si: $\mathrm{I} \xrightarrow{5°,0.4\text{ GPa}} \mathrm{I+II} \xrightarrow{5°,0.6\text{ GPa}} \mathrm{I+II+III} \xrightarrow{82°,8.3\text{ GPa}} \mathrm{I+II+III+XI} \xrightarrow{12\text{ GPa}} \mathrm{II+XI} \xrightarrow{0\text{ GPa}} \mathrm{XII+III}$ (Cu)
100 nm Si: $\mathrm{I} \xrightarrow{2.2\text{ GPa}} \mathrm{I+II} \xrightarrow{31.8°,4.2\text{ GPa}} \mathrm{I+II+III} \xrightarrow{0\text{ GPa}} \mathrm{I+II\&I+III\&I+II+III}$ Rough anvils (S.S.)
100 nm Si: $\mathrm{I} \xrightarrow{9.1°,3.6\text{ GPa}} \mathrm{I+II} \xrightarrow{0\text{ GPa}} \mathrm{I}$ Rough anvils (S.S.)

New rules for coupled severe plastic deformation, strain-induced phase transformations, and nanostructure evolution under high-pressure

Dr Feng Lin[1], Professor Valery I Levitas[1], Dr Pandey Krishan[2], Dr Sorb Yesudhas[1], Dr Changyong Park[3]
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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Study of the plastic flow, strain-induced phase transformations (PTs) [1], and microstructure evolution, as well contact friction under high-pressure is important for producing new nanostructured phases [2] and understanding physical and geophysical processes [3]. However, these processes depend on an unlimited combination of five plastic strain tensor components and an entire strain path with no hope of fully comprehending. Here [4], we introduce the rough diamond anvils (rough-DA) to reach maximum friction equal to the yield strength in shear, which allows determination of pressure-dependent yield strength. We apply rough-DA to compression of severely pre-deformed Zr.

We found in situ that after severe straining, crystallite size and dislocation density of α and ω-Zr are getting pressure-, strain- and strain-path-independent, reach steady values before and after α-ω PT, and depend solely on the volume fraction of ω-Zr during PT. Immediately after completing PT, ω-Zr behaves like perfectly plastic, isotropic, and strain-path-independent with the fixed surface of perfect plasticity in the stress space. Friction stress is also getting plastic strain- and strain-path-independent. Rough-DA produce a steady state in α-Zr with lower crystallite size and larger dislocation density than smooth diamonds. This leads to a record minimum pressure (0.67 GPa) for α-ω PT with rough-DA, much smaller than 1.36 GPa with smooth diamonds, 6.0 GPa under hydrostatic condition, and phase equilibrium pressure, 3.4 GPa. Existence of the multiple steady states and, consequently, surfaces of perfect plasticity, all strain- and strain-path-independent, causes the major challenge in plasticity theory. The kinetics of strain-induced PT, in addition to plastic strain, unexpectedly depends on time. This opens an unexplored field of the simultaneous strain- and stress-induced PTs under pressure. The obtained results create new opportunities in material design, synthesis, and processing of nanostructured materials by severe plastic deformations at low pressure. Rough-DA can be utilised for finding similar laws for various material systems. The above plethora of results was obtained in a single experiment, thus transforming the main challenge – strongly heterogeneous fields in a sample – into a great opportunity.

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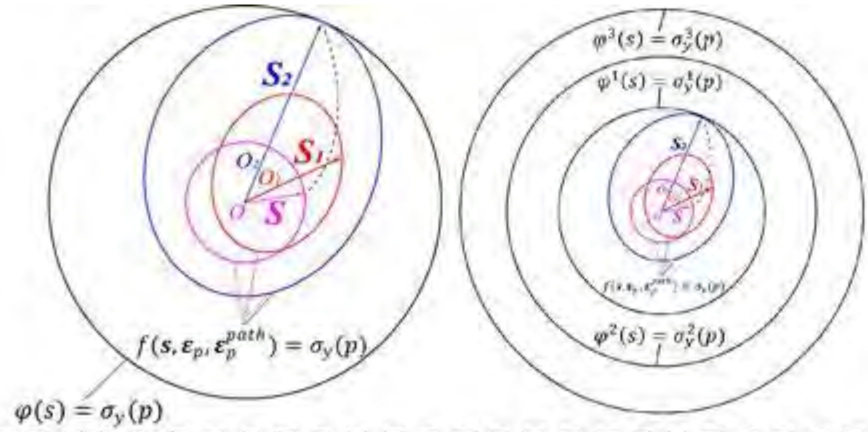
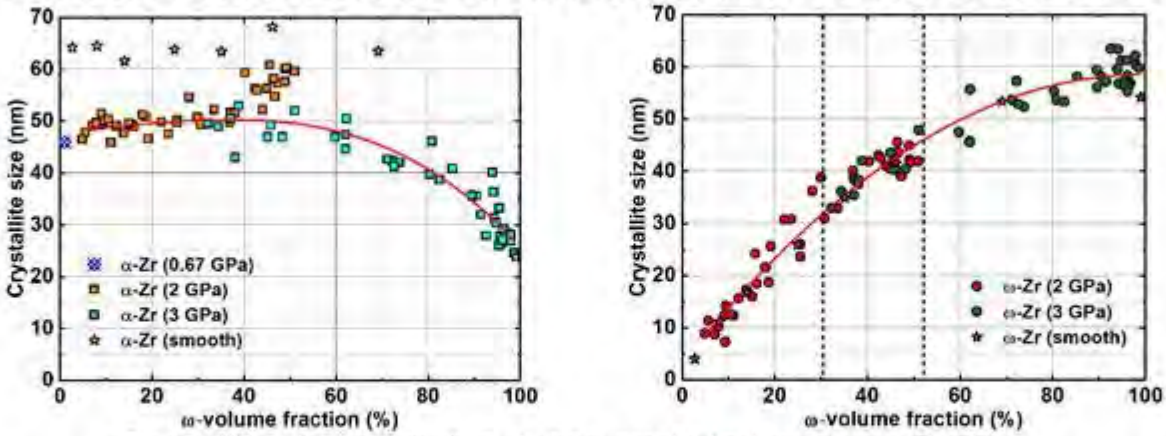
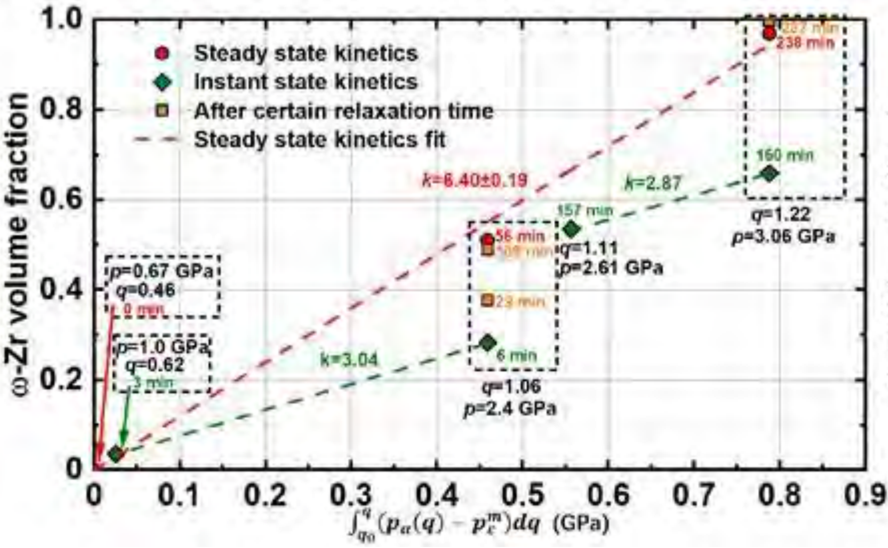


Evolving yield surface and multiple surfaces of perfect plasticity



Crystallite size evolution during strain-induced α-ω Zr PT



Time dependence of strain-induced α-ω PT kinetics.

Zero order kinetics independent of volume fraction.

Tensorial stress-plastic strain fields in α- ω Zr mixture, transformation kinetics, and friction in diamond anvil cell

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Various phenomena (phase transformations, chemical reactions, microstructure evolution, strength, and friction) under high-pressures in diamond anvil cell are strongly affected by fields of all components of stress and plastic strain tensors. However, they could not be measured. Even measured pressure distribution contains significant error because axial strain does not contribute to X-ray patterns, and the equation of state determined under hydrostatic conditions is used for nonhydrostatic loading. We suggest coupled experimental-analytical and experimental-analytical-computational approaches utilizing synchrotron X-ray diffraction to solve an inverse problem and find fields of all components of stress and plastic strain tensors in each phase and mixture and friction rules before, during, and after α-ω phase transformation in strongly plastically predeformed Zr.

Our results demonstrate that both approaches are in good correspondence with each other and experiments, which significantly advances the understanding of material behaviour. Due to advanced characterization, the minimum pressure required for the strain-induced α-ω phase transformation is changed from 1.36 to 2.7 GPa, independent of the plastic strain before phase transformations and compression-shear path. The theoretically predicted plastic strain-controlled kinetic equation is verified and quantified. Our findings open opportunities for developing quantitative high-pressure/stress science, including mechanochemistry, synthesis of new nanostructured materials, geophysics, and tribology. This study emphasises the importance of advanced characterization techniques and the need for a multifaceted approach to study material behaviour under high-pressures.

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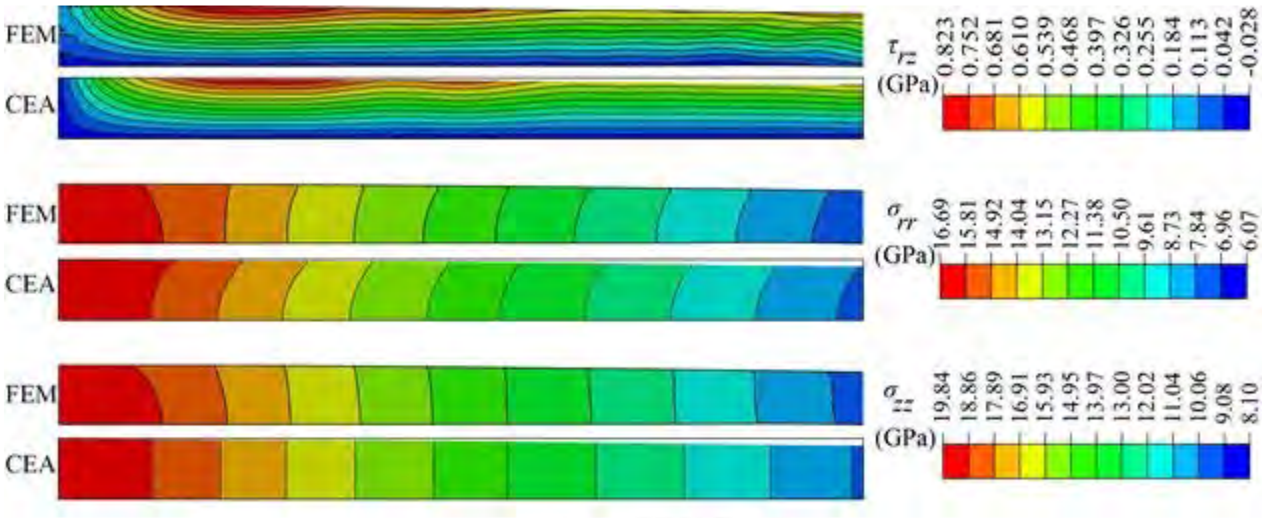
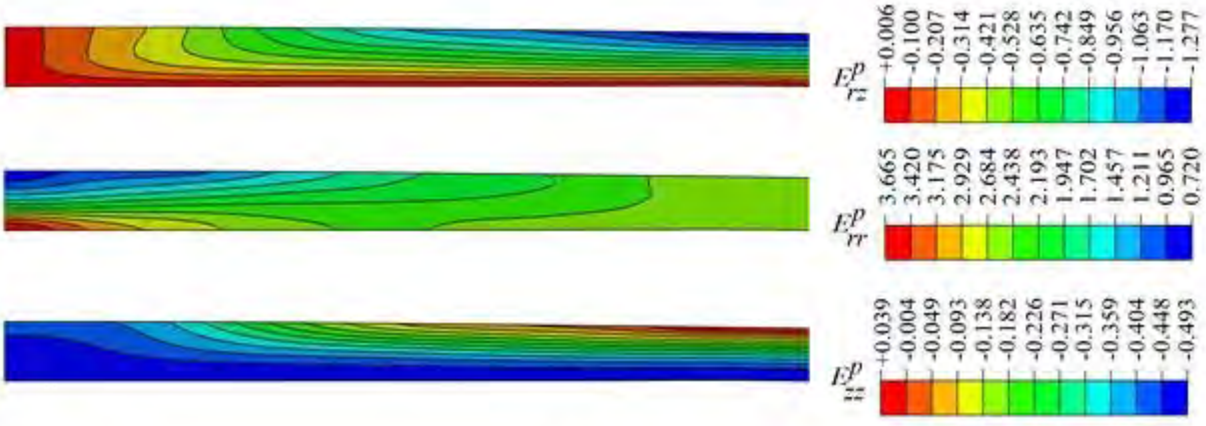


p_{max} = 17.55 GPa



p_{max} = 17.55 GPa

Present and Future Extreme Conditions Research at Low (PETRA III) & Ultra-Low (PETRA IV) Emittance Synchrotron Sources at DESY

Hanns-Peter Liermann[1], Konstantin Glazyrin[1]
[1]DESY, Hamburg, Germany, [2]DESY, Hamburg, Germany

Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

With the advent of high energy ultra-low emittance synchrotron sources extreme conditions research at high-pressure and high/low temperatures has enter a new realm. While at 3rd generation sources mostly static high-pressure experiments have been performed, the 4th generation sources will enable the exploration of hierarchical structures both in space and time, making optimal use of the low emittance and even more important the superb coherence of these new sources.

In this presentation we demonstrate the current status of DAC research capabilities at the Extreme Conditions Beamline P02.2 at PETRA III and high-light the new developments. Particular emphasised will be the combination of time resolved X-ray diffraction and coherent imaging that has been developed at P02.2 in order to study hierarchies in space and time, which will be the major focus of the Extreme Conditions Time Resolved XRD & Imaging Microscope (ExTReM) at PETRA IV. Thus, ExTReM will combine the unprecedented brilliance and coherence of PETRA IV to perform static (min) to fast (MHz) high-P and high/low-T X-ray diffraction and imaging experiments at high energies to create the ultimate microscope that enables the study of 5 dimensions spanning 6–7 orders of magnitude in time, spatial resolution, and compression rate.

In situ X-ray diffraction of the α-ε phase transition in iron at intermediate strain rates

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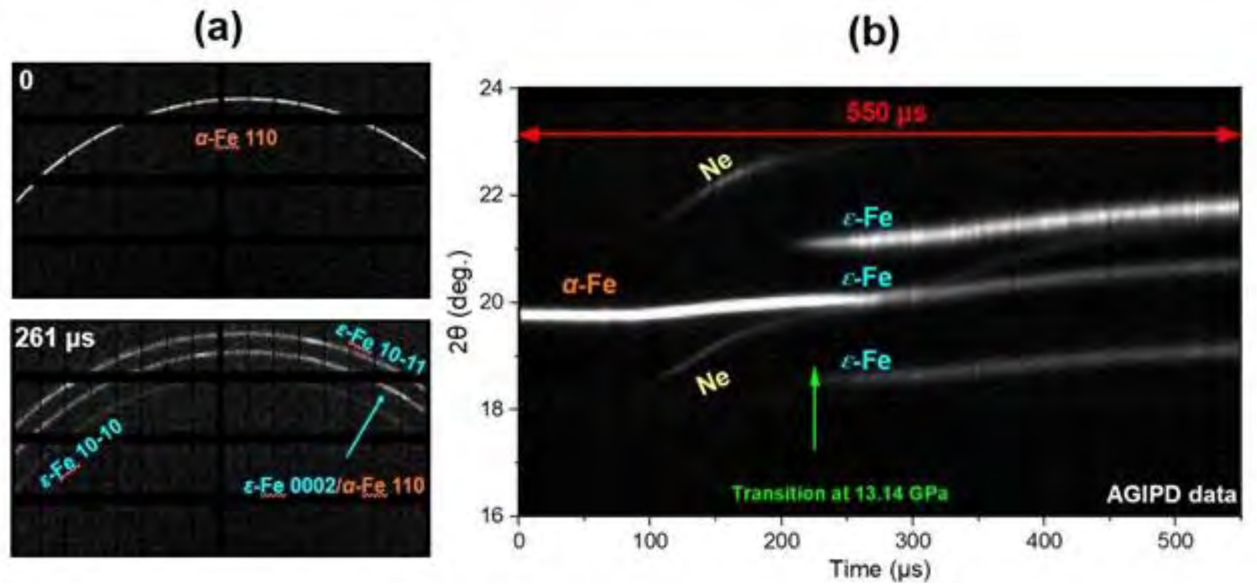
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The α(bcc)-ε(hcp) phase transition of polycrystalline Fe has been investigated under fast compression using the dDAC platform at the High Energy Density (HED) Instrument at the European X-ray Free Electron Laser (European XFEL). Taking the advantages of the unique long X-ray pulse-trains and high repetition rate detector, we are able to collect the pulse-resolved X-ray diffraction data of Fe under intermediate strain rate compression ($\leq 10^3$ s^(-1)) at MHz repetition rate. Our results demonstrate for the first time the full process of α-ε phase transition of Fe under dynamic compression at intermediate strain rates. Under fast compression (~70 TPa·s^(-1)), the α-ε phase transition of Fe is likely to be fully transformed in a time scale of tens of microseconds with a pressure interval of about 2–3 GPa, and it seems that the phase transition pressure of Fe is relatively little affected by the strain rate at low pressure.



Superionicity of hot dense fcc ice evidenced by X-ray diffraction

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Since decades, calculations predicted superionic (SI) states in water ice at extreme pressures and temperatures [1–4]. In the SI state, protons diffuse in a liquid-like fashion throughout a solid oxygen sublattice. Two phases reported as SI were recently observed in shock wave and static experiments [5–9]. One phase shares the bcc oxygen sublattice of ice VII and X, while the other has a fcc oxygen sublattice. The fcc phase appears to have a very large stability field in the high-pressure P-T phase diagram but large discrepancies exists between both static and dynamic studies. In addition, existing experimental hints of superionic fast proton conduction are only based on optical measurements and remain elusive [5,8].

We used in-situ synchrotron X-ray diffraction to study water ice under various conditions (P = 30–200 GPa and T = 300-2600 K) in a laser-heated diamond anvil cell. A new sample confinement was implemented with two opposite boron-doped diamond laser absorbers forming a heating capsule in which the ice sample is confined. Coupled to the two-sided laser heating setup at the ID27 beamline of the ESRF, it enabled to heat a large volume of ice with reduced thermal gradients hence to collect high quality data.

Our measurements show that the bcc-fcc transition plateau at 2000 K in agreement with Ref [9]. Moreover, above 80 GPa, hot fcc ice phase can be quenched down to ambient temperatures.
By measuring the volume of the fcc phase at various pressures (50-200 GPa) and temperatures (300–2600 K), we could evidence a jump of the thermal expansion. The latter is identified as a Schottky-bump signature of the superionic transition (see figure 1), a well-documented mechanism in superionic conductors such as fluorites [10].

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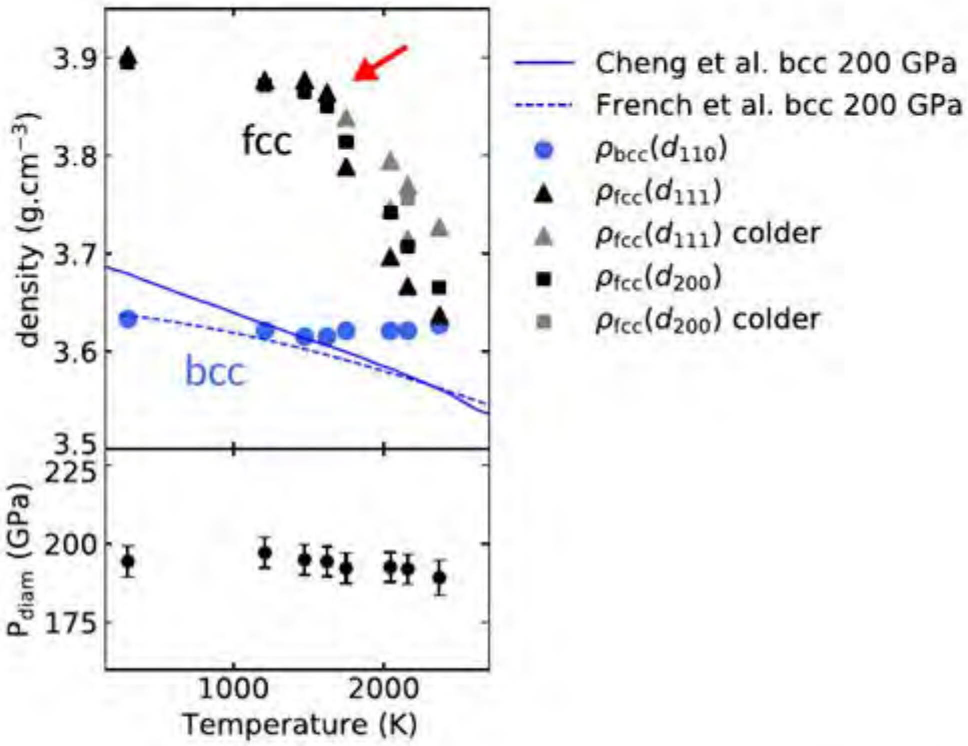

Figure 1 : Temperature evolution of the bcc and fcc ice densities at ~193 GPa and high temperatures, showing a high thermal expansion regime of Schottky bump (red arrow). Pressure measurements via XRD peaks of the boron-doped diamond absorber are included in the bottom panel, showing no pressure drop during the heating ramp. Grey points corresponds to colder fcc crystals due to thermal gradients.

Pressure-tuned blue-to-orange light emission in polymorphic diphenylmaleic anhydride

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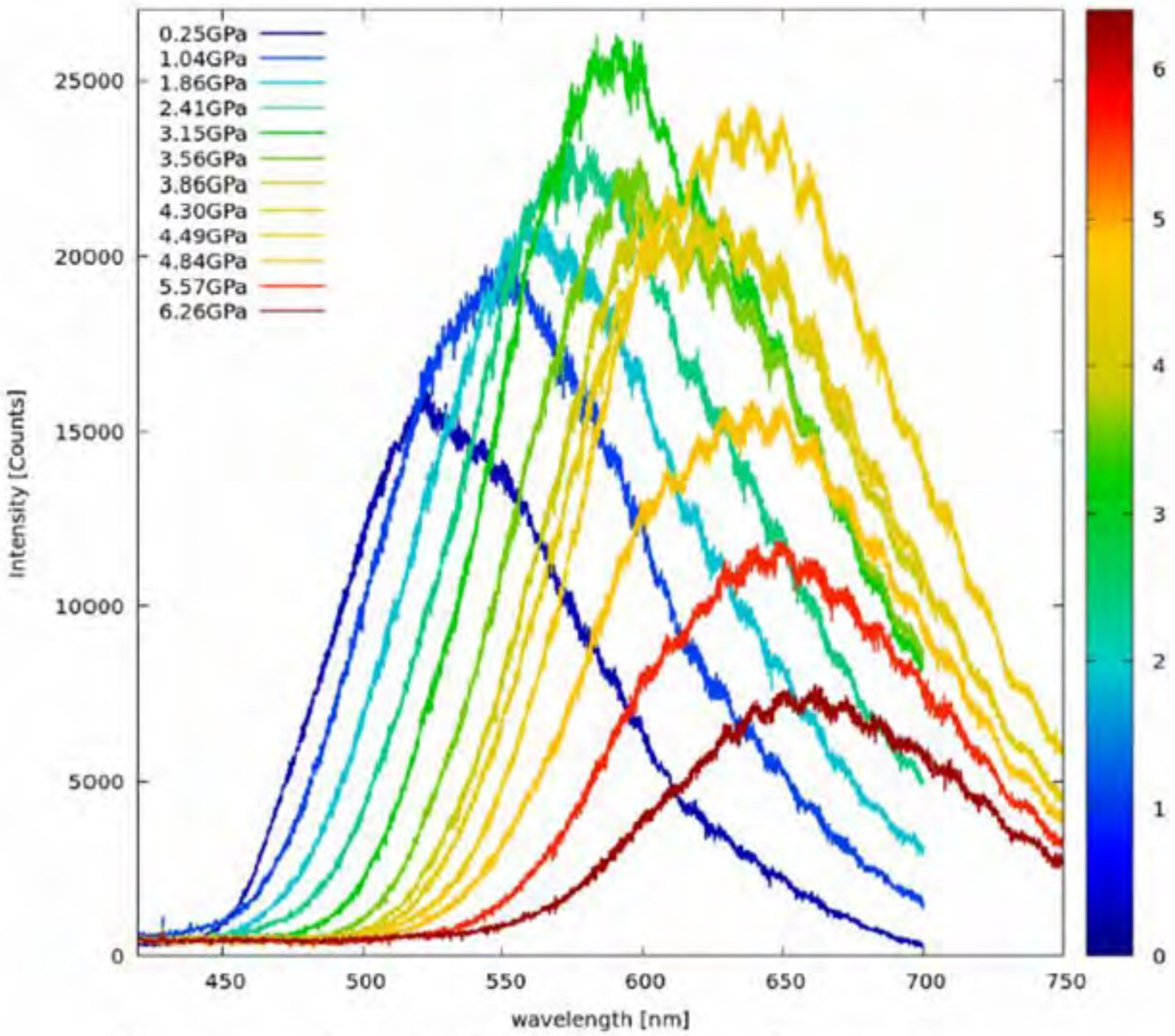
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

2,3-diphenylmaleic anhydride (DPMA) presents aggregation-induced emission in its two polymorphic forms. In this work, we investigate an effect of pressure on the structure and luminescent properties of both polymorphs.

They show a strong luminescence around 480 nm at ambient conditions. As the pressure increases, the intermolecular envelope shrinks, promoting non-radiative pathways through enhanced pi-pi stacking. However, the extend of this behaviour is different in two forms, due to differences in packing, despite the similar pressure-induced red-shift rate (530 nm at around 3.5 GPa). Irradiation of the solution of DPMA with UV light causes electrocyclization. Such reaction does not occur in solid state due to steric hindrance imposed by the neighbouring molecules, bound mainly with carbonyl-carbonly interactions. The condensate presents, unlike parent molecule, phenantrene-like planar geometry. The crystals of condensate show remarkable red-shift to upon compression from 500 nm (ambient pressure) to 660 nm at circa 6 GPa.



Fluorescence change in a 2,3-diphenylmaleic acid condensate (phenantrene anhydride) upon compression

Melting curve of black phosphorus and associated colossal volume jump

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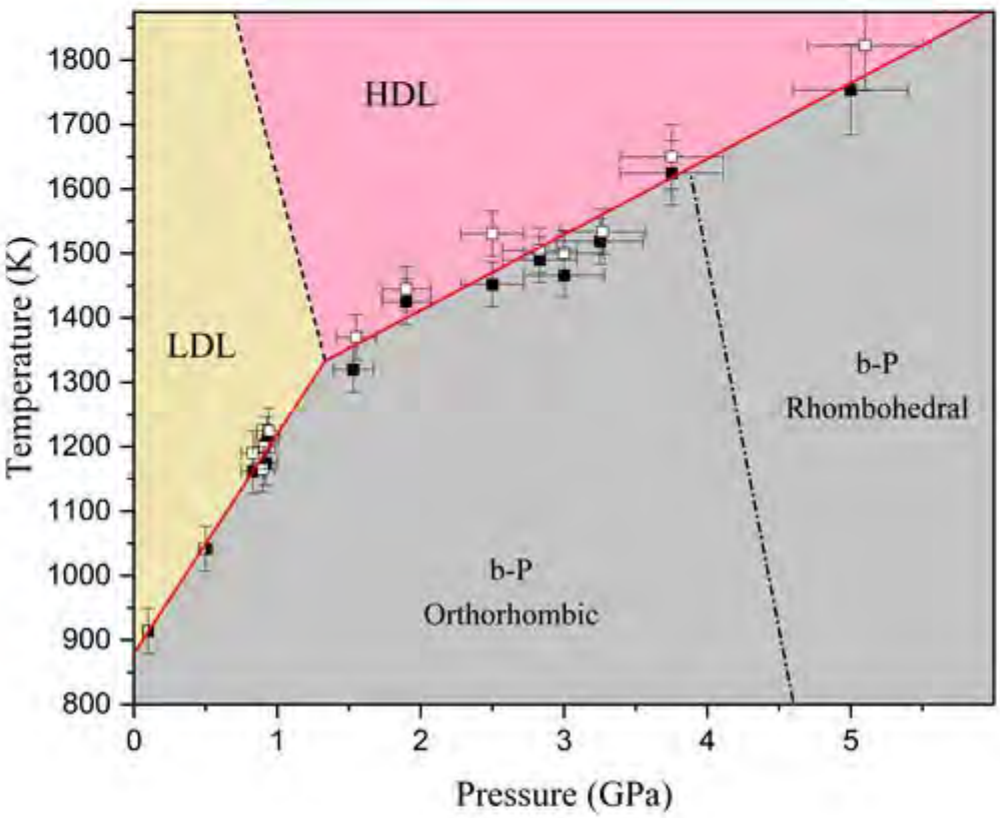
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Phosphorus exhibits a very rich polymorphism under ambient but also under high P and T conditions. Among them, black phosphorus (bP) occupies a very special place as the most stable allotrope in a wide P,T region of its phase diagram. Due to the exceptional anisotropic properties of solid bP and the existence of a first-order liquid-liquid transition, this element has been extensively studied. However, several important questions remain open regarding its liquid state and melting line. In particular, it is expected that significant changes occur on the melting curve and on the liquid-liquid transition (LLT) in the vicinity of the liquid-liquid-solid triple point. However, there are still large discrepancies in the literature regarding its shape and position. In order to redetermine the controversial melting curve of bP and extend the determination of the LLT, as well as the density change across these transitions, we performed combined in situ X-ray diffraction and density measurements.

We report the revisited melting curve of bP showing a dramatic slope change at 1.33 GPa and 1340 K. We report the evidence for a solid-liquid triple point where the slope change occurs. Surprisingly, bP has been found to be reversible only in the pressure region above the pressure of the solid-liquid triple point, while red phosphorus is recovered when quenching from the low-density liquid. Density measurement across the melting line have showed unprecedent results: a colossal volume variation is observed as bP melts to the low-density liquid phase (up to 150% volume variation at the transition), denoting the dramatic structure change undergoing at the transition. This is to our knowledge the most dramatic volume variation ever observed for the melt of a single-component material. From the slope of the melting curve and the volume variation at the transition, the calculated entropy of melting of ≃70 J.K-1.mol-1 was obtained. On the other hand, a volume variation of less than 10% is observed when melting towards the high-density liquid, resulting in an entropy variation at the transition of ≃ 5 J.K-1.mol-1. These results suggest that the high-density liquid structure is very close to the solid structure of bP, while the low-density liquid resembles red-P.

We have extended the determination of the LLT of bP up to 2500 K and measured colossal density variations across the LLT, similar to the melt toward the low-density liquid. 175% volume variation was observed at 2500 K. The result is in good agreement with MD simulations, and no critical point was observed up to 2500 K. The critical point of bP could lie above this temperature but we expect this point to be located at negative pressures since the LLT presents a negative slope.


HDL
LDL
b-P
Rhombohedral
b-P
Orthorhombic
Temperature (K)
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
0
1
2
3
4
5
Pressure (GPa)

TiC-MgO composite: an X-ray transparent and machinable heating element for the multi-anvil high-pressure apparatus

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Experiments conducted under simultaneous high-pressure and high-temperature conditions are necessary for studying Earth's deep interiors and for characterizing the properties of materials under extreme thermodynamic conditions. Multi-anvil apparatus can nowadays generate pressures over 1Mbar with homogeneous temperature field, and, thus, is a suitable device for high-pressure study. However, the bulky sample environment imposed by experimentation in large volume press has been limiting the number of usable techniques and the quality of the data. In particular, X-ray based observations are primary tools to characterise the properties of materials in situ at high-pressure and high-temperature conditions. The combination of multi-anvil apparatus and in situ X-ray observations requires X-ray transparent heating elements. Graphite, which is both refractory and X-ray transparent, is an excellent heating element but only at pressures lower than 10 GPa as above graphite converts to diamond and can't be used as a heating element anymore. To overcome this hindrance, TiB2-BN composite or boron-doped diamond have been proposed as X-ray transparent heating elements. However, TiB2-BN composite heater is limited in stable temperature generation (< 2000 K), and boron-doped diamond is inconvenient in fabrication due to its extreme hardness. In this study, a TiC-MgO composite was developed as a heating element for in situ X-ray experiments in the multi-anvil high-pressure apparatus.

We synthesised TiC-MgO blocks (50–70 wt.% of TiC) by compression in a cold isostatic press followed by baking in a gas flow furnace. Heaters of tubular shape were manufactured from the synthesised blocks either by lathe or numerically controlled milling machine. With this heater, we have succeeded in generating temperatures up to 2250 K stably at pressures above 10 GPa and successfully conducted challenging in situ X-ray radiography and diffraction measurements on liquid Fe alloys. Being cheap, easy to fabricate, X-ray transparent, and stable in heating, the TiC-MgO heater is promising to be a routine heating element for multi-anvil experiments.

New insights in chemical and mechanical effects in Ru(II)-Ru(III) Bonds

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Usually, the $\sigma < \pi < \delta < \delta^* < \pi^* < \sigma^*$ orbital ordering is assumed for multiple metal-metal bonds. [1] However, this is defied by diruthenium mixed-valence paddlewheel compounds, which present a quasi-degeneracy of their π^* (dxz-dxz; dyz-dyz) and δ^* (dxy-dxy) orbitals. Most of these complexes present three unpaired electrons in their ground state, which is typically represented with the $(\pi^*\delta^*)^3$ electronic configuration. [2] Nevertheless, the closeness in energy of π^* and δ^* gives rise to a borderline stability between $S = 3/2$ and $S = 1/2$ states that is strongly dependent on the ligand field. New insights on how these orbitals are affected by changes in the nature of the ligands or pressure can pave new ways to control their magnetic and electronic properties.

In this work, we present our studies regarding changes in the equatorial ligand field due to distortions of the paddlewheel structures. These are provoked mainly by changes in the functional groups in the aromatic rings of the ligands, and by the application of external pressure. Whereas increasing the Leq-M-M-Leq torsion angles is known to elongate M-M distances due to a weaker δ-overlap, [2] our synthesised crystal structures of Ru_2^5+ complexes with different equatorial ligands show the opposite behaviour. Unfortunately, modifications in the aromatic substituents are needed to change the Neq-Ru-Ru-Neq torsion angle, which implies that electronic and torsion effects are always coupled. Through absorption and Raman spectroscopy experiments under high-pressure, we show how it's possible to uncouple these effects, thus providing new insights on how distortions affect bond distances and orbital energies in diruthenium compounds.

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Effect of the incommensurate Bi-III phase on the Bi-Sb system under pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The high-pressure phase behaviour of the Bi–Sb system was investigated experimentally and modelled thermodynamically to study the effect of the transition to the incommensurate Bi-III type structure, which is adopted by the high-pressure phases Bi-III and Sb-II of the pure elements.
The thermodynamic model of the Bi–Sb phase diagram including the effect of pressure in the isomorphous regime was extended to include solid–solid phase transitions of the pure elements. Consequently, the alloy phase diagram was found to transform to the eutectic form with pressure and is predicted here in the pressure range of 3–6 GPa. The alloy was found to transform from the ambient, rhombohedral A7 structure to the incommensurate phase. The transformation occurred at intermediate pressures between those observed in elemental Bi and Sb. The region of thermodynamic stability was determined by calculating the spinodal curves. To verify these predictions, the structure of a Sb-rich alloy was determined as a function of pressure by X-ray diffraction measurements in a diamond anvil cell, demonstrating the transition from A7 to the incommensurate Sb-II-like structure. It was found that the decomposition of the alloy under pressure is determined by the thermodynamic instability located at the spinodal curve.
In contrast, alloy compositions located in the metastable region between the binodal phase line and the spinodal curve did not decompose under pressure, thus indicating a new region of phase space to be explored in high-pressure studies of alloys.

Single crystal structure refinements and Debye temperatures of kashinite-bowieite (Ir2S3-Rh2S3) and erlichmanite-laurite (OsS2-RuS2) solid-solutions

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The kashinite-bowieite (Ir2S3-Rh2S3) and erlichmanite-laurite (OsS2-RuS2) solid-solutions crystallise in the early sequences of mineral crystallisation differentiation from the primitive magma in the Earth's mantle. The structures of natural and synthesised crystals were refined using single-crystal X-ray diffraction analysis. The kashinite-bowieite solid-solution has a unique sesquisulfide structure which is isostructural with the high-pressure form of Rh2O3. The erlichmanite-laurite solid-solution has the pyrite-type structure. The bulk Debye temperatures were estimated from the Debye-Waller factor for the sulphide site. The bulk Debye temperatures of OsS2 and RuS2 were 688 K and 661 K, respectively. The bulk Debye temperatures for these minerals were found to rank among the higher values reported for many known sulphides. The high Debye temperature of OsS2 is inconsistent with the crystallisation of laurite prior to erlichmanite from the primitive magma, which suggests that f(S2), rather than temperature, is the main cause of the known crystallisation order. Crystallisation of these minerals in Earth's mantle was discussed from the obtained Debye temperatures.

High-pressure synthesis and characterization of NiAs-type novel manganese mononitride

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Transition metal nitrides are attracted attention in material physics and chemistry fields. Especially, nitrides synthesised under high-pressure have unique crystal structures and nitrogen-rich compositions that can be synthesised only under high-pressure, and their elastic properties and electronic structures are attracting attention. The crystal structures of high-pressure nitrides have high-density, highly coordinated polyhedra, nitrogen dimers or polymeric nitrogen, and distinctive properties. For instance, FeN and CoN [1] have highly coordinated crystal structures and ferromagnetic order, and IrN_2 [2] and ReN_2 [3] having nitrogen dimers have high bulk modulus comparable to diamond. These crystal structures and properties depend on synthetic pressure and the kind of transition metal atoms. Thus, to understand these interesting characteristics of transition metal nitrides, more investigations are required. In this presentation, we report the characterization of novel manganese mononitride, NiAs-type MnN synthesised through a pressure-induced phase transition from tetragonally distorted MnN around 55 GPa by using a laser-heated diamond anvil cell. The in-situ X-ray diffraction measurements revealed the phase transition of MnN from NiAs-type structure to tetragonally distorted NaCl-type by decompression. The compression behaviour for NiAs-type MnN was also revealed by the in-situ X-ray diffraction measurements. In addition, we will report the detailed phase stability, compression behaviour, and magnetic property of NiAs-type MnN were investigated by high-pressure in-situ synchrotron X-ray diffraction measurements and a first-principles calculation.

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Probing iron's spin state in FeS at conditions of the Martian core

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The novel approach of combining a diamond anvil cell sample environment with X-ray heating and MHz-resolved X-ray diffraction (XRD) measurements has been recently introduced to the High-Energy-Density (HED) instrument of the European XFEL (EuXFEL) [1]. It enables measurements of the samples' structure at static high-pressures (p) and extreme temperatures (T) and, in future, also at unique thermodynamic conditions induced by e.g. laser shock compression [2].

We used the von Hámos spectrometer implemented in the interaction chamber 1 of the HED instrument, which is dedicated to X-ray emission spectroscopy (XES) measurements at extreme p/T conditions. We exploited the X-ray heating as applied in a successful XES proof-of-principle experiment [3,4] to the Fe-S system. The sample represents a simplified composition of the Martian core, which consists of molten Fe-Ni with up to 15wt% S and 5 wt% O [5].

We measured spin-sensitive Fe Kβ XES spectra from FeS at conditions close to p/T of the Martian core [6], including FeS in the melt state. The temperatures and changes in the samples' structure were monitored with simultaneously measured XRD, allowing us to interpolate the spin state development throughout the probed p-T field. The results suggest a spin state change within the FeS-IV and FeS-V stability field between 700-1800K and 6-25GPa from an initial low-spin state at ambient temperatures to a complete high spin state of Fe.

Acknowledgements: We acknowledge European XFEL in Schenefeld, Germany, for providing XFEL beamtime at the Scientific Instrument HED (Proposal #2804) and would like to thank the staff for their assistance. We thank C. Schmidt from GFZ Potsdam and H.-P. Liermann from the PETRAIII beamline P02.2 for supporting the sample characterization. We are grateful to the HIBEF UC for the provision of staff to execute the experiment. This work was supported by the BMBF via 05K19PE2.

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Thermal conductivity of deep Earth minerals and alloys measured at extreme pressure-temperature conditions

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Measurements of thermal conductivity of the Earth's mantle minerals and core alloys at extreme pressure-temperature conditions (P-T) provide important insights into planetary dynamics and thermal history. Careful estimations of thermal conductivity of deep Earth, especially near the core-mantle boundary (CMB), requires better knowledge of the effects of pressure, temperature, composition, spin state, and grain size on the thermal conductivity values of the major Earth's minerals and alloys. Here I present an update of the laser spectroscopy techniques which we developed to measure conductive and radiative thermal conductivities as well as emissivity of materials at extreme P-T conditions in laser heated diamond anvil cells (DAC). Transient heating technique to measure the conductive part has been recently greatly improved by using the fibre laser pulse of a rectangular shape using a Pockels cell. This and other technical modifications increased the accuracy of thermal conductivity values obtained in our finite-element calculation of the heat transport in a DAC. Moreover, in collaboration with a high-pressure community, we developed transient heating experiments at the European X-ray Free-Electron Laser (EuXFEL) in Hamburg (EuXFEL Proposal 3160, Co-PIs R. S. McWilliams, and C. Sanchez-Valle), where the samples of interest in a DAC are being heated by a train of X-ray pulses absorbed by the material and deposited heat sequentially into the bulk of the sample. The evolution of the sample temperature with time is being measured via the thermal expansion probed by instantaneous XRD measurements using the same X-ray pulses and streaked optical pyrometry (SOP). Thermal conductivity of the material is determined by how fast the system reaches the steady state. Recent results of investigation of ferropericlase, bridgmanite, mineral mixtures with pyrolite composition and Fe alloys will be presented shedding new light on the heat transport in the mantle and the core and thermal history of the Earth.

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Electrical resistance of Fe_2O_3 at high-pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

It is essentially important to estimate the generated pressure in high-pressure experiments. Pressure calibration by using pressure fix points indicated by a change in electrical resistance of a material is useful method to estimate pressure in the opaque apparatus such as a Kawai-type multianvil apparatus (KMA) installed in the laboratory but not in synchrotron facility. Although we have been able to generate pressure nominally more than ~30 GPa due to the recent development of hard material for the anvils, it is difficult to confirm the generated pressure in the laboratory because the pressure fixed-point has been limited to ~30 GPa (e.g., 34.5 GPa for Zr [1]). Ito et al. [2] suggested that the electrical resistance of Fe_2O_3 decreases significantly at ~58 GPa, but the details are unknown.
In this study, therefore, we focus on Fe_2O_3 because it is considered to be a candidate of calibrant and moreover it may be useful analogue as A_2O_3 compound for Earth's mantle material. In this study, we investigated the phase transformation and the electrical resistance of Fe_2O_3 under high-pressure and high temperature. We conducted in-situ X-ray observation at high-pressure and high temperature in the KMA up to ~60 GPa with the measurements of electrical resistance for sintered aggregate of Fe_2O_3. The measurements were done by the (pseudo) 4-wire method to allow us to measure very low resistance. At the experiment, we investigated the temperature dependences of electrical resistance of hematite at ~15 and 34 GPa and obtained the large activation enthalpies to be ~45 kJ/mol (~0.46 eV) and ~34 kJ/mol (~0.35 eV), respectively. Then, hematite transformed into Rh_2O_3-type structure at ~47 GPa and high temperature as reported by previous workers [2,3], and activation enthalpy was as same order of magnitude as those of hematite at lower pressure and it was determined to be 22 kJ/mol (~0.23 eV). By subsequently compression, further transformation to perovskite-like structure (phase III) was observed at ~51 GPa and high temperature. The activation enthalpy of this phase was determined to be ~2 kJ/mol (~0.02 eV) which is much smaller than those at lower pressure phases. This suggests that the electrical structure of perovskite-like phase is largely different from the other phases. On the other hand, the electrical resistance at room temperature is largely decrease when dominant phase was Rh_2O_3-II phase without apparent phase transition, suggesting the isostructural electrical transition in Rh_2O_3-II phase. Although additional experiment to clarify the phase present, our preliminary results suggest that it is not simple relationship between the electrical resistance change and phase transformation.

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High-pressure Raman study of hcp metals Be, Os, and Re up to 200 GPa

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Hexagonal close packing (HCP) is a common crystal structure of many important metals with some interesting properties, such as anisotropy, high melting point, etc. In terms of material application and scientific nature, it is of great significance to study the properties and behaviour of HCP metal under extreme conditions. Such as the most incompressible metal, osmium (Os), whose high-pressure behaviour is studied by high-pressure X-ray diffraction has attracted much interest and arguments for its possible electronic topological transition （ETT） under compression. However, as an effective in-situ measurement method, Raman scattering could provide vibrational information inside the crystal, supporting the precise detection of such pressure-induced structural changes. But Raman spectroscopy in HCP metals at high-pressure was restricted by the technology challenges including the strong anvail diamond fluorescence and the terrible signal associated with the high reflectivity of metal surface in compressed sample cavity. As a result, the high-pressure Raman spectroscopy of HCP metallic elements is not well understood at the pressure above 150 gigapascals.

Here we report Raman scattering measurements with the lateral excitation technology with 45◦ incidence angle on HCP metals beryllium (Be), osmium (Os), and rhenium (Re) using diamond anvil cell (DAC) at high-pressures up to 73 GPa, 220 GPa, and 200 GPa, respectively. We also designed a simple Raman spectroscopy technique for the HCP metals under high-pressure, which could obtain a comparative effective signal improvement effect. The pressure-dependent shear modulus C44 of those metals has been deduced from the Raman-active mode E2g, providing valuable elastic properties information under high-pressure. In addition, we study the lattice dynamics behaviour of metal Os under high-pressure in the pressure range of 0~220 GPa. The analysis showed that the pressure coefficient（dω/dP）of E2g Raman mode in Os was mutated at ~110 GPa and the mode-Grüneisen parameter produced a corresponding sudden drop, revealing a possible ETT of Os at high-pressure.

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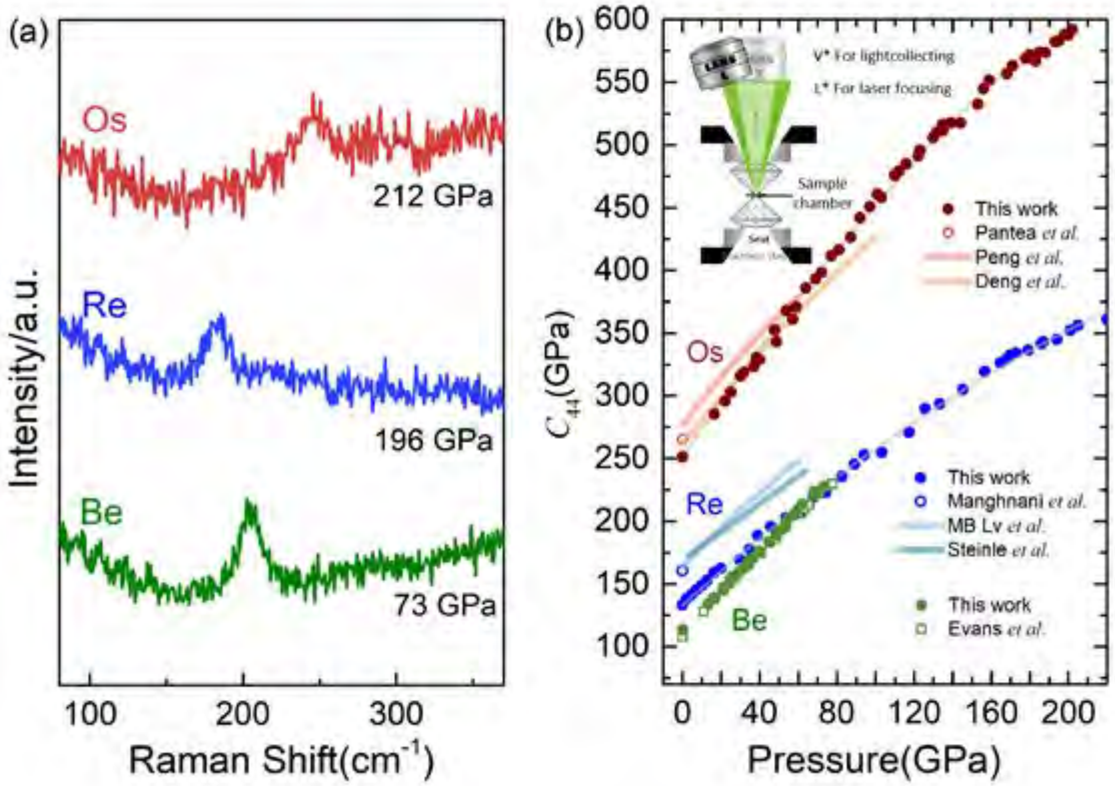
(a)
Os
212 GPa
Re
196 GPa
Be
73 GPa
Intensity/a.u.
100
200
300
Raman Shift(cm^{-1})
(b)
V* For lightcollecting
L* For laser focusing
LENS
Sample chamber
Seat
This work
Pantea et al.
Peng et al.
Deng et al.
Os
Re
Be
This work
Manghnani et al.
MB Lv et al.
Steinle et al.
This work
Evans et al.
C_{44}(GPa)
100
150
200
250
300
350
400
450
500
550
600
0
40
80
120
160
200
Pressure(GPa)

$Pr_3Mo_4B_6O_{24}(OH)_3$: High-Pressure/High-Temperature Synthesis of an Acentric Rare Earth Molybdenum Borate

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Around 1980, first rough investigations of rare earth molybdenum borates were reported, but information about these compounds is rather scarce [1,2]. In the years 2008 to 2011, progressing synthesis methods for these compounds not only led to the single-crystal structure re-determinations of these compounds, but also disclosed their excellent optical properties. A potential application in self-frequency doubling laser devices or as high-performance LED-phosphors were discussed recently [3–5]. This work focuses on new compounds within this promising class of substances, synthesised under high-pressure/high temperature conditions in a modified Walker-type multianvil device.
We report on the synthesis of a new praseodymium molybdenum borate under high-pressure/ high-temperature conditions. The new compound with the sum formula $Pr_3Mo_4B_6O_{24}(OH)_3$ crystallises acentrically in the monoclinic space group Cm displaying a new structure type. The structure was solved via single-crystal structure determination. Additionally, the proposed structural model was confirmed by powder X-ray diffraction, second harmonic generation measurement, and single-crystal infrared and Raman spectroscopic investigation. For further information see https://doi.org/10.1002/ejic.202200635.

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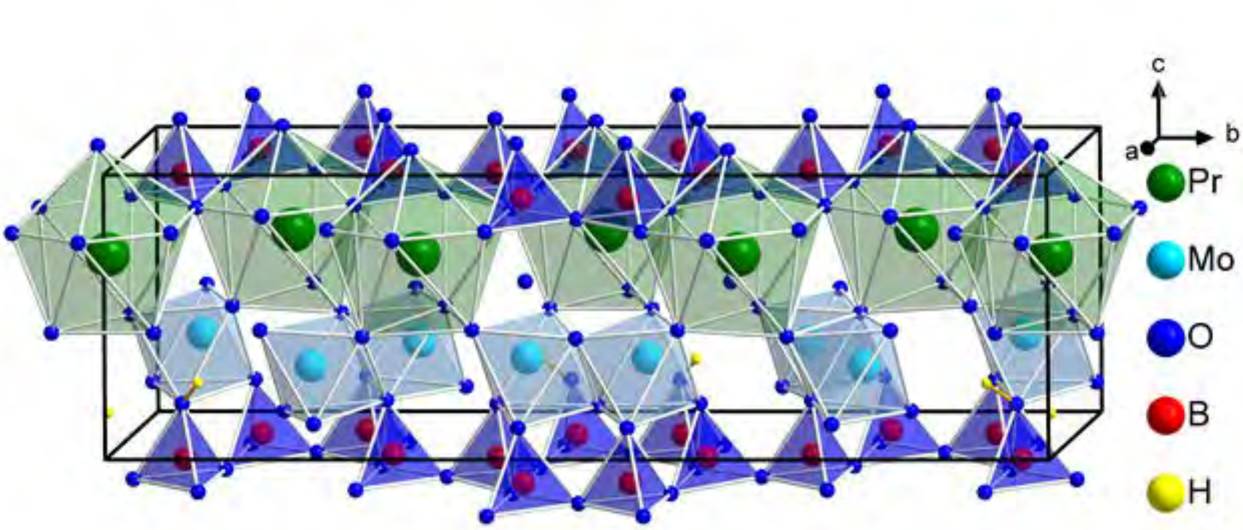
c
a
b
Pr
Mo
O
B
H

Synthesis and crystal structure of the zinc borate Zn3B4O9

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Borates are an interesting substance class because of their structural diversity. This diversity originates from the different tetrahedral BO4 and trigonal planar BO3 units and exceeds the diversity of silicates. Some crystalline borates are suitable as host material for luminescent ions like Eu3+ in YBO3 [1] or Mn2+ in Zn4O(BO2)6. [2] High-pressure/high-temperature conditions further expand the reachable chemical space enabling the synthesis of new compounds and structures. At a pressure of around 10 GPa and a temperature of 1173 K, the new zinc borate Zn3B4O9 forms, exhibiting an isotypic structure to Sr3G4O9 [3], which is synthesizable at normal pressure. Zn3B4O9 crystallises in the space group $P\bar{1}$ (no. 2) with a = 5.5028 (2) Å, b = 6.7150 (3) Å, c = 7.8887(3) Å, α = 83.99 (1)°, β = 73.38 (1)°, γ = 74.75 (1)°, V = 269.35 (2) Å3, and two formula units (Z = 2) per unit cell. It shows a step-like layered structure and can be doped with Mn2+ to give a red luminescence after UV excitation.

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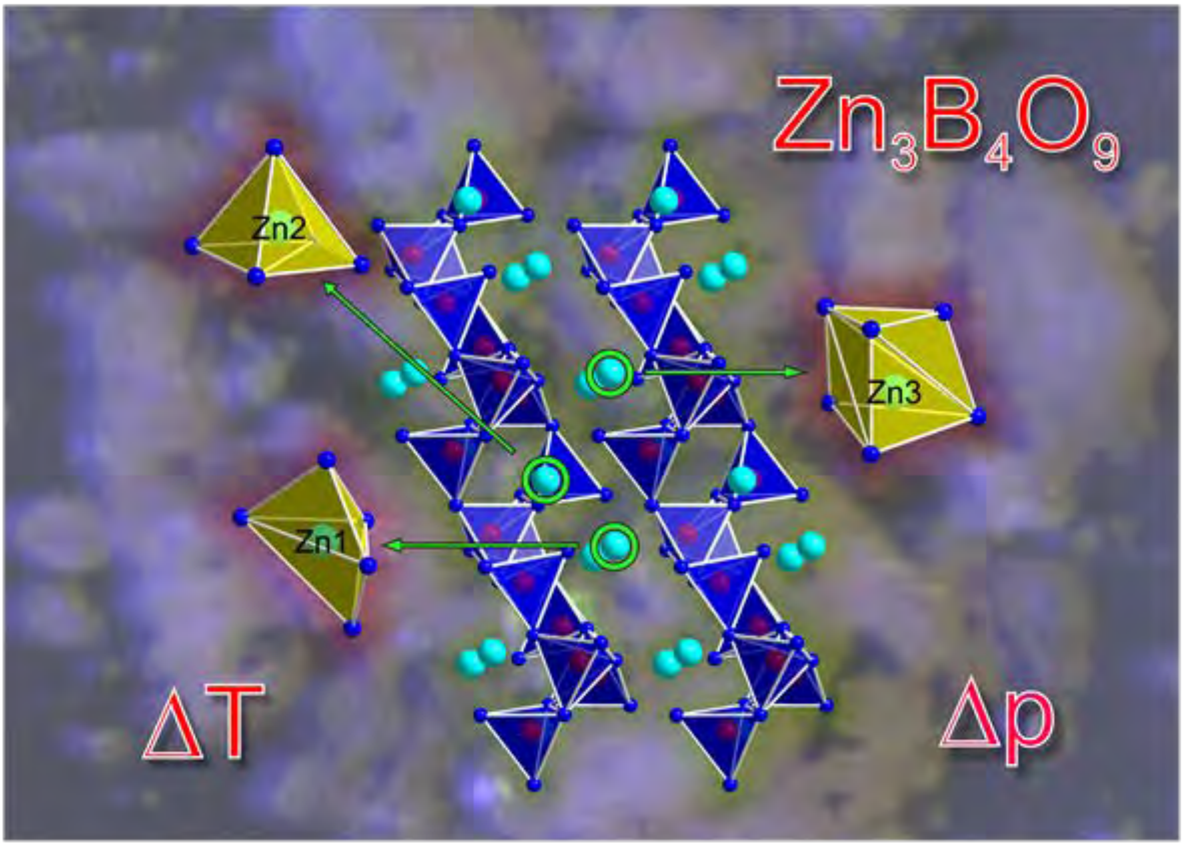

Xpress beamline, Diffraction at Extreme Conditions – Elettra Sincrotrone Trieste

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The Indo-Italian high-pressure (HP) diffraction beamline, Xpress, at Elettra Sincrotrone Trieste, is increasing its' operational performances by adding new instruments. The beamline utilises the synchrotron radiation from a superconducting wiggler to produce a 25 keV monochromatic X-ray beam focused on a large area detector for data acquisition in angle dispersive mode. This configuration allows powder and single crystal (SC) diffraction experiments to be performed under HP conditions by using diamond anvil cells (DAC), comprising an online fluorescence spectrometer (ruby, $Sm^{2}+:SrB_4O_7$). The beamline station is equipped with state-of-the-art facilities for HP manipulation: microscope, microdriller, automatic pneumatic pressure controller, etc. The partner institute, IISc Bengaluru, recently added three new dedicated HP setups compatible to DAC: (i) a recently commissioned low temperature (T) closed cycle He-cryostat, reaching down to 10 K (Almax easyLab/ JANIS), see Fig. 1; (ii) still on commissioning (under improvement): a high T vacuum chamber, making use of internal and external heater, reaching up to 1000 K (LOTO-eng); and (iii) a recently commissioned micro Raman spectrometer (Renishaw, class 4). On the top of this, the beamline team, in collaboration with the Elettra's Research Engineers (RE) group, had in house developed: 1) a cryogenic loader to permit the loading of a variety of gas media, such as Ar, N_2, O_2, CH_4, etc.; and 2) a second vacuum chamber to be used for HP-high T experiments by using an external ring heater (1000 K). The later vessel is on the final assembling stages and compatible for in situ synchrotron XRD measurements. Still on new loading possibilities, a commercial gas loading system (Sanchez) was recently commissioned, which permits DAC to be loaded with gases such as He, Ne, etc. Beamline data collection and control graphical user interface is being continuously improved by the Elettra's Information Technology group to handle well all these new developments even on remote mode.

Elettra is currently undertaking an upgrade program, Elettra 2.0, to reach an even brighter and coherent synchrotron radiation source. Under this project, Xpress beamline recently received a new, large area detector, DECTRIS PILATUS3 S 6M (see Fig. 1). Which key advances are: direct detection of X-rays; single-photon counting; excellent signal-to-noise ration and very high dynamic range; short readout time and high frame rates; shutterless operation; modular detectors enabling multi-module detectors with large active area. As a consequence, not only reducing significantly the HP diffraction data collection time, but also increasing the Q-range spectra and data quality if compared to the previous detector (MAR345). Still under the Elettra 2.0 upgrade program, Xpress very recently obtained a space dedicated to loading and HP manipulation: Elettra's High Pressure Lab. Xpress developments are in progress, perspectives for a near future are powder and SC diffraction under HP (up to megabar pressures) together with variable temperatures from few to several hundred Kelvins. Proposal submission deadline are typically on mid-March and -September.
We will present recent scientific highlights from the beamline performed under extreme conditions of pressure and temperature.

Figure 1. Closed cycle He-cryostat and PILATUS3 S 6M detector.

Hyperbaric storage of human milk at sub-zero temperature – impact on microbiota, leukocytes and basic nutrients

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

According to WHO recommendations, breastfeeding is the best method of feeding new-borns and children up to two years of age. The composition of human milk (HM) is perfectly adapted to the needs of developing children at each stage of their growth and is one of the main factors responsible for maintaining their good health into adulthood. In the case of premature babies, breastfeeding ensures the proper maturation of the underdeveloped digestive and immune systems and significantly reduces the risk of certain diseases. Therefore, when a mother cannot breastfeed, the best alternative, especially when feeding premature infants, is to use HM pasteurised and stored in Breast Milk Banks (HMB). Heating HM at 62.5°C for 30 min leads to a significant reduction in its nutritional and biological value. Therefore, new methods of preserving human milk are being searched for that will ensure its microbiological safety and maintain its health-promoting properties as much as possible. The purpose of this study was to determine the effect of hyperbaric storage at 60 MPa/-5°C and 111 MPa/-10°C on the survival of human milk microbiota, concentration of leukocyte, macronutrient, and energy value. It was found that the total bacterial count (TBC) in the HM was 4.6 log CFU/mL, while the number of coagulase-positive staphylococci was 4.1 and 4.7 log CFU/mL in batch A and B, respectively. Enterobacteriaceae and Bacillus cereus were not detected in the tested milk. After 7 days of HM storage at 60 MPa/-5°C, TBC decreased by 1.6 log cycles, while at 111 MPa/-10°C bacteria were not detected. Complete reduction of TBC in milk stored at 60 MPa/-5°C was obtained after 63 days. Staphylococcus aureus cells were very sensitive under these conditions and were not detected after 7 days of hyperbaric storage. TBC and S. aureus counts were also reduced in samples stored at -5 and -10°C under atmospheric pressure. The coagulase-positive staphylococci were not detected after 7 days at 0.1 MPa/-5°C, and after 21 days at 0.1 MPa/-10°C. The content of leukocytes in HM stored at sub-zero temperature both at atmospheric and moderate pressure decreased gradually to about 2 log cycles. Hyperbaric storage at sub-zero temperatures for 63 days did not cause changes in the content of total protein, nutritional protein, and carbohydrates of HM. A statistically significant higher total fat content and energy value was noted in samples stored under elevated pressure than at 0.1 MPa. In conclusion, hyperbaric storage at sub-zero temperature allows for the inactivation of human milk microbiota without significant negative changes in its nutritional value.

Novel Material Synthesis @ Large Volume Press Beamline P61B PETRA III

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The Large Volume Press (LVP) 'Aster-15' is installed and operational for user experiments at high-pressures and temperatures (HP-HT) at the PETRA III P61B beamline [1]. P61B is the end-station of the high-energy wiggler beamline at PETRA III. X-ray availability is equally shared between P61A (Helmholtz-Zentrum Hereon) and P61B-LVP beamline station. Therefore, in addition to in-situ energy-dispersive X-ray diffraction (EDXRD) between 30 and 160 keV at HP-HT conditions (routinely up to 25 GPa and 2000 °C), offline LVP time (without X-rays) is also available for users to perform HP-HT experiments. Users are offered the chance to conduct in-situ experiments that primarily aim to uncover a new phase or material, while offline experiments can then be utilised to replicate the results on a larger scale under the necessary HP-HT conditions.

Here we report a typical example of in-situ tracking and ex-situ synthesis; HP-HT synthesis and characterization of the structural and mechanical properties of a γ-Si_3N_4/Hf_3N_4 ceramic nanocomposite derived from a single-phase amorphous Si-Hf-N precursor. The structural evolution of the amorphous precursor and its crystallisation to γ-Si_3N_4/Hf_3N_4 nanocomposites under high-pressure is assessed by in-situ EDXRD measurements at ~19.5 GPa in the temperature range from ca. 1000 °C to 1900 °C. The final synthesis of the γ-Si_3N_4/Hf_3N_4 nanocomposite is performed at ~20 GPa and ca. 1500 °C using 'Aster-15' in an offline mode.

Our poster will also present further offline synthesis efforts at P61B, including a newly discovered 'Nolanite-type' phase of tin-germanium oxy-nitride (Sn-Ge-O-N) and recently reported novel materials like tin oxy-nitrides (Sn_2N_2O) [2] and ternary silicon titanium nitrides (Si-Ti-N) [3]. The complexity of synthesizing a large volume of material under HP-HT conditions and the challenges involved in characterizing multi-phase compounds are demonstrated in our studies.

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High-pressure study of PbGa2S4

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Lead thiogallate, $PbGa_2S_4$, belong to the wide class of chalcogenide compounds of general formula AII $BIII_2CVI_4$ and some representatives of these compounds with defect chalcopyrite and defect stanite structure at room pressure, such as (Cd, Hg)Ga_2S_4 and (Zn, Cd, Hg)Ga_2Se_4, have been thoroughly studied at high-pressures [1]. $PbGa_2S_4$ crystallises at room conditions in the $EuGa_2S_4$-type orthorhombic (space group Fddd) structure with unit cell parameters a = 20.706, b = 20.380, and c = 12.156 Å [2]. Up to our knowledge, there is no high-pressure study of $PbGa_2S_4$ or any isostructural AII $BIII_2CVI_4$ compound, despite they could be useful for various nonlinear optical devices and optical filters as evidence the papers that have investigated the properties of $PbGa_2S_4$ at room pressure [2–8]. In this work, the results of the analysis of Raman Scattering, X-Ray Diffraction, and optical absorption measurements under pressure will be shown. Evidences of a pressure-induced phase transition will be also commented.

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Synchrotron-based structural characterization of Sr2FeIrO6 under high-pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Perovskites are one of the most extensively studied materials in Solid State Physics. The ideal perovskite crystallises in cubic structure with ABX_3 stoichiometry, where the large cation (A atom) is surrounded by 12 X atoms while the smallest cation (B atom) lies in octahedral coordination. The size of the BX_6 octahedron can be tuned by changing the A atom, due to its property of expanding/ shrinking and tilting to compensate the size of the A polyhedral units. Furthermore, the B atoms play an in important role by dominating the electrical and magnetic properties. The replacement of one cation B by exactly half of the number of atoms of other element (from now on, atoms called B' and B'') lead to the formation of the so-called double perovskites with a stoichiometry $A_2B'B''O_6$.

This work presents results on the high-pressure behaviour of Sr_2FeIrO_6 compound. This material has generated some controversy in the literature with the assignation of its most stable structure. Originally, the ambient phase was assigned to a I-1 (sg. 2) structure.[1] This very asymmetric phase was due to a certain cationic disorder by the exchange between the Sr and Fe atoms. Nevertheless, a recent article shows that the distorted structure does not tolerate the cationic disorder,[2] triggering a phase segregation between a stoichiometrically pure phase forming a I-1 (sg. 2) arrangement and another doped or disordered phase with a $P2_1/n$ structure (sg. 14). Additionally, previous analysis claims that the original phase belongs to the I2/m space group (sg. 12). [3]

DFT-theoretical calculations are insufficient to solve the ambiguity of the ambient pressure phase due to the small difference of Enthalpy energy among both tentatively assigned original phases. In order to elucidate the original phase of the Sr_2FeIrO_6 compound, the bibliography shows a clear sequence of phases applying high temperature to the double perovskites. For instance, in the case of Ba_2-xSrxYRuO_6 [4] or Sr_2NiIrO_6 [10] is Fm-3m→I4/m→I2/m→$P2_1/n$. This sequence can be used as a guideline to determine the starting phase with the help of the phases observed. Thus, this work investigates on the results obtained in similar compounds and takes advantage of Synchrotron-based high-pressure and high-resolution X-ray diffraction measurements to evaluate the ambient structure and the possible phase transitions.

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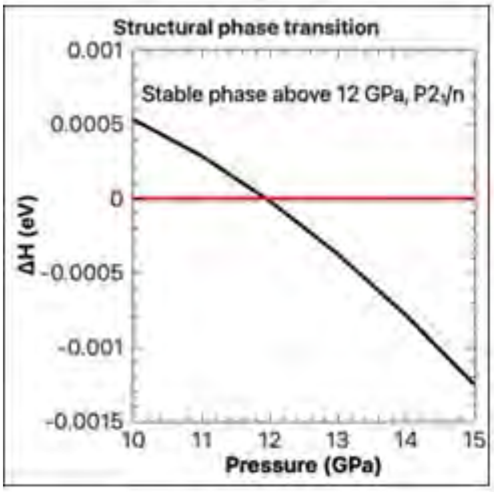
Structural phase transition
Stable phase above 12 GPa, P2₁/n
ΔH (eV)
0.001
0.0005
0
-0.0005
-0.001
-0.0015
10
11
12
13
14
15
Pressure (GPa)


2.4 GPa
4
6
8
10
12
14
16
2theta (degrees)

Equations of state of metals (Fe, Mo, Cu, W, Re, Pt, Au) and magnesium oxide (MgO) at multi-megabar pressure

Takeshi Sakai[1], Hirokazu Kadobayashi[2], Yuusuke Kara[3], Shota Masuda[3], Hideto Mimori[1], Yuki Nakamoto[3], Keitaro Kuramochi[4], Naoki Ishimatsu[4], Saori Kawaguchi[2], Miyu Karita[1], Yuki Tanaka[3], Yusuke Seto[5], Yuki Kato[3], Soutaro Iwatsu[3], Yoshiki Kenmochi[1], Satoru Nakamura[1], Naohisa Hirao[2], Oki Sekizawa[2], Kiyofumi Nitta[2], Katsuya Shimizu[3], Takehiko Yagi[6]
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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

In high-pressure experiments, the pressure value is estimated from the unit cell volume of pressure standard materials using its equation of state. Various metals, oxides, and alkali halides are often used as pressure standard materials, as they have large X-ray scattering power and simple crystal structures that are stable over a wide pressure range. Each primary pressure scale should be mutually consistent, and the simultaneous volume measurement in static compression enable us to check the consistency using the volume-volume relation. The unit cell volumes of metals and/or oxide in Cu-Re, Pt-Re, Cu-W, Au-W, Mo-W, Fe-W, MgO-W, MgO-Pt combinations were measured simultaneously using toroidal diamond anvil cell (t-DAC) and/or laser-heated DAC (LHDAC) at multi-megabar condition. Culet sizes of 10–20 um were used for t-DAC experiments. The unit cell volumes were measured by the powder X-ray diffraction measurement at SPring-8 BL10XU and BL37XU. The volume data is obtained up to around 300–400 GPa for each experiment, and maximum pressure was reached to 450-480 GPa in run for Cu-Re, although the pressure value depends on which pressure scale was adopted. Present volume-volume relations provide information on the intercomparison of each equation of state, and allows us to construct internally consistent pressure scales for a single pressure scale and to discuss the consistency between primary scales. Present results indicate that there is still about 5% pressure mismatch between the recently proposed primary scales of Pt, Au, and Cu under multi-megabar pressure conditions.

Thermal conductivity of H2O ice VII from X-ray heating experiments at the European XFEL

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Water (H2O) ice is a major constituent in many astronomical bodies, such as comets and asteroids, or the interior of the solar ice giants (Neptune and Uranus) and mini-Neptune exoplanets [1,2]. H2O has an interesting phase diagram with up to date 20 known solid phases, two superionic ices and various amorphous structures [3,4]. The body-centred cubic ice VII phase is stable over a wide range of temperatures and pressures and its thermal conductivity is an important parameter to model the structure and thermal evolution of these (exo)planets. Difficulties arise, however, when an attempt is made to measure this quantity at these extreme conditions (>50 GPa, >1000 K) due to limited experimental techniques. As a result, the thermal conductivity of Ice VII was only investigated up to 22 GPa at room temperature up to date [5]. Here we present an original approach to determine the thermal conductivity of planetary materials, including H2O ice, using the ultrafast (MHz) X-ray pulse trains of the European XFEL to simultaneously heat and probe the sample inside a diamond anvil cell. The experiments were conducted at the HED (High Energy Density) station within the community proposal 3160. The thermal expansion can be determined using time resolved X-ray diffraction in the time span of 550 µs and combined with finite element modeling, the heat flow can be characterised. We present and discuss new results on the thermal conductivity of H2O ice VII at higher pressures and temperatures up to 20-65 GPa and 900 K that supersede the previously known data [5].

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High-pressure structural stability and luminescence studies of nanoclays for environmental applications

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Clays are widely used nanomaterials with important environmental applications. They are proposed as part of the engineered barrier system (EBS), as the ideal backfill material to immobilise high-level radioactive wastes (HLW) in deep geological repositories (DGR). Under this premise, clay minerals are situated around the containers with the purpose of retarding the diffusion of nuclear wastes through the geosphere and sealing the tunnel and boreholes [1]. The preference for nanoclays to be used as a component of the EBS in DGR stems from their desirable engineering properties, such as low hydraulic conductivity to water, kw (typically < 10-10 m/s), and the existence of semipermeable membrane behaviour, the latter giving rise to hyperfiltration, chemical-osmotic flow, and reduced diffusion [2]. Also, nanoclays are important components of the more eco-friendly water-based drilling fluids [3]. Drilling fluids are pumped down the well controlling formation pressures, removing cuttings from the borehole, cooling and lubricating the bit, and maintaining the wellbore stability, among others [4]. In both cases, the long-time behaviour of these plate-like particles is affected by the temperature and pressure generated in the underground by the working conditions. Here, we present a study of the effects of hydrostatic pressure on the structural stability and optical properties of a set of synthetic nanoclays, proposed for those particular environmental applications. In particular, the nanoclay Laponite has been studied by X-ray diffraction and Raman spectroscopy to evidence possible new high-pressure phases and to determine its volume compressibility, paying special attention to the loss of its layered structure. Also, Eu3+ doped high-charge nanoclays have been synthesised and Eu3+ luminescence has been measured to investigate changes in the crystalline structure of the nanoclay under pressure. In this way, Eu3+ has been assessed as structural probe and used as a model to study the effect of the bidimensional confinement in the interlayer cation under extreme conditions.

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High-pressure synthesis of multi-components (Cr1-x-yMnxVy)P4 phosphides

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

MP_4 materials were first synthesised and explored in the 1970s using high-pressure techniques, however, it did not attract much attention. Recently, a boom in black phosphorus and its related 2D layer structure compounds brought back the interest in MP_4 materials. Black phosphorus is famous because of its outstanding thermal, electrical, optical, and tuneable band structure properties, and has been expected to be a candidate of 'beyond graphene' material. For black phosphorus, the characteristic 2D structure is in analogue to graphene. The transition metal phosphides with the layer black phosphorus structure have thus become a focus for understanding and exploring novel material properties.

Under high-pressure it was predicted that black phosphorus would become topological semimetal, a superior candidate for high performance electronic devices. Interestingly, recent high-pressure experiments in transition mental phosphides - CrP_4 [1] and MoP_4 [2], which consist of the black phosphorus layer, also show topological semimetal characteristics. Being said, other phosphides with similar black phosphorus layer structure may as well show exceptional electric property.

Herein, in searching for a new phosphorus system with the black phosphorus layer structure, we combined multi-components (multiple cation species) into the system of MP_4. In past studies, VP_4 [3] and CrP_4 [4] were reported to have the same crystal structure and MnP_4 was found to be the stacking variant of CrP_4 [5]. They all have black phosphorus stacking layers. In our experiments, multi-components $(Cr1-x-yMnxVy)P_4$ compounds are synthesised under high-pressure ($P \leq 5$ GPa) and high-temperature ($T \leq 1200\ ^{o}C$) using large volume press, the recovered products are then characterised using synchrotron XRD, SEM-EDS and SQUID.

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Stability and High-Pressure behaviour of Paracetamol polymorphs through Raman spectroscopy

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Paracetamol or Acetaminophen (C₉H₈NO₂) is one of the most relevant drugs, commonly used due to its antipyretic and analgesic properties. Like many other drugs, it presents different structural conformations stable at ambient conditions, which can be associated with useful properties for the pharmacological industry, <i>e.g.</i> tableting [1].

Up to nine Paracetamol polymorphs have been reported so far, and six of them can be stabilised at ambient conditions (Forms I, II, III, VII, VIII, and IX). Two high-pressure polymorphs, Forms IV and V, have been also reported, although their structure and properties remain unknown [2]. Paracetamol Form I (S.G. <i>P2₁/a</i>) undergoes a structural phase transition to Form II (S.G. <i>Pcab</i>) at around 6 GPa, showing a narrow pressure phase coexistence as determined by Raman spectroscopy [2]. Form II has been investigated by XRD showing no phase transition in the 0–5.5 GPa range [3], although a transition to the Form IV polymorph (structure unknown) has been reported on powder samples when starting from the Form I polymorph [4]. Additional high-pressure studies on Paracetamol starting from the Form II polymorph have not been reported so far [1].

In this work, we study the high-pressure structural evolution of Form II powdered Paracetamol by Raman spectroscopy in the 0-25 GPa range, including Form I samples and Form I/II mixtures for comparison purposes. The temporal stability of the Form II polymorph at ambient conditions and the effects of composition and pressure transmitting medium on the structural stability of Form II have been also analysed.

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Crystal structure and high-pressure phase behaviour of a CaCO3-SrCO3 solid solution

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Carbonatites and carbonate inclusions in diamonds and mantle minerals are characterised by a rich variety of compositions, pointing to the importance of extending the study of carbonates to more complex systems other than pure end-members. A synthetic CaCO3-SrCO3 solid solution, with composition Ca0.82Sr0.18CO3, recovered from a high-pressure, high-temperature experiment (2 GPa, 1273 K), has been recently investigated by Raman and Fourier transform infrared spectroscopy up to 40 GPa and 800 K [1], showing how the cationic substitution of Ca by Sr has a drastic effect and modifies the phase diagram of pure CaCO3. Here, we present the results from single crystal X-ray diffraction (XRD) studies performed on samples from the same synthesis batch as [1] up to ~9 GPa using Ne as pressure transmitting medium (PTM) and up to ~20 GPa in Ar PTM to better understand the structural evolution of Ca0.82Sr0.18CO3 under pressure. At ambient conditions the material has a monoclinic symmetry, isostructural to CaCO3-II, with space group P21/c and 4 formula units per unit cell (Z), named Sr-calcite-II (Sr-CC-II). The unit cell parameters are: a = 6.4237(7) Å, b = 5.0176(1) Å, c = 8.1129(1) Å, β = 108.064(1)° and V = 248.60(1). At 1.72(5) GPa, Ca0.82Sr0.18CO3 undergoes a pressure-induced structural transition to a monoclinic structure (space group P21/m and Z = 8), named Sr-calcite-III (Sr-CC-III), which was observed up to the maximum experimental pressure of 9 GPa in Ne PTM. The unit cell parameters at 1.72(5) GPa are: a = 6.2683(2) Å, b = 9.9220(5) Å, c = 7.6574(6) Å, β = 103.856(6)° and V = 462.39(5) Å3. This high-pressure structure is characterised by non-coplanar CO3 groups and different cationic sites with coordination number of 7 and 8.

This structure is different from any known polymorph of end-member CaCO3. At 12 GPa Sr-CC-III transforms to a triclinic structure, Sr-calcite-IIIb (Sr-CC-IIIb), space group P-1, Z=4 and unit cell parameters: a=6.059(5) Å, b=6.280(2) Å, c=6.331(2) Å, α=95.20(3)°, β=108.89(5)°, γ=110.52(5)° and V=207.7(2) Å3. At 17 GPa Sr-CC-IIIb transforms to a new triclinic polymorph, Sr-calcite-VI (Sr-CC-VI) with space group P-1, Z=2 and unit cell parameters: a=3.444(3) Å, b=4.985(4) Å, c=5.761(5) Å, α=77.05(7)°, β=84.92(7)°, γ=89.00(7)° and V=96.0(1)Å3. Overall, the substitution of Ca2+ with the larger cation Sr2+ causes a distortion of the calcite structure at ambient conditions and the crystallisation of new structures at high-pressures different from those observed in end-member CaCO3.

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Structural changes in hydrous sodium silicate melts under high-pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Water is the major volatile component in magma, and the density [1] and viscosity [2, 3] of magma strongly depend on its water content. These physical properties vary greatly depending on the chemical composition of the magma (silicate melt) and the depth (pressure) of the Earth's interior, which is strongly related to changes in the melt structure (e.g. [4]). However, there are few studies on the structure of hydrous silicate melts [5], and the effects of water content and pressure on melt structure have not been well understood. In this study, we chose a hydrous sodium silicate $Na_6Si_8O_{19}$ (N3S8) melt, whose water solubility has been investigated by previous spectroscopic experiments [6], and performed in situ X-ray diffraction and neutron diffraction measurements under high-pressure to clarify structural changes in the melt.

We successfully obtained the total structure factors of dry and hydrous N3S8 (containing 9-12 wt% water) melts up to 7 GPa. First sharp diffraction peak (FSDP) positions of the total structure factors related to the intermediate-range order structures shift to high Q-region with increasing pressure, indicating that the network structure based on the SiO_4 tetrahedra shrinks under pressure. Both pair distribution functions (PDF) derived from the X-ray and neutron total structure factors show that the Si-O bond distance of SiO_4 tetrahedra in dry N3S8 melt is almost constant at pressures up to 7 GPa, whereas the Si-O bond distance increases in hydrous N3S8 melt. The results indicate that the Si-O-Si bond angle does not change with pressure because hydrous N3S8 melt is compressed by a different mechanism from dry N3S8 melt.

It is well known that the addition of water to silicate melt breaks the bonds between the SiO_4 tetrahedra due to the formation of hydroxyl species via Si-O-Si + H2O → 2·Si-O-H. The PDF analyses also show that the O-H(D) bond distance in hydrous N3S8 melt increases with pressure, while the O-O bond distance decreases. In addition, ab initio molecular dynamics simulations reveal that the number of O-H species in hydrous N3S8 melt decreases with pressure, while the number of H_2O molecular species remains constant. Therefore, these results suggest that in the compression of hydrous N3S8 melt, the polymerization of the network structure and the increase -O-H-O- bridging species through the reaction Si-O-Na + Si-O-H → Si-O-H-O-Si + Na+ are preferred over narrowing of the Si-O-Si bond angle.

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ScAlO3 perovskite under high-pressure from first principles simulations

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

ScAlO3 perovskite can be considered as a prototype of ABO3 perovskite structure with a strong analogy with MgSiO3. At low temperatures and pressure, ScAlO3 has an orthorhombic structure with Pbnm symmetry. In this work, we present an ab initio study of this compound under high-pressure. We report the evolution under hydrostatic pressure of the structural, dynamic, and elastic properties of the low-pressure phase of this compound. The evolution under pressure of infrared, silent, and Raman frequencies and their pressure coefficients are also presented. The dependence on the pressure of the elastic constants and the mechanical and elastic properties are analysed. We also propose a candidate as a high-pressure phase for this compound and we report the structural, dynamic, and elastic properties of this high-pressure phase.

Lattice dynamics and heat capacity of multilayer graphane

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Multilayer graphane (hydride of graphite) is a new hydrocarbon of composition CH, synthesised at high hydrogen pressure at ISSP RAS [1]. Using X-ray diffraction, it was preliminarily identified as the “graphane II” phase of 3D-graphane predicted by ab initio calculations [2] and consisting of layers of 2D-graphane in the chair conformation stacked along the hexagonal c-axis in the -ABAB- sequence. The calculations also showed that in the phonon density of states g(E) of the chair modifications of graphane, there is a gap between the acoustic and optical bands. This provides the opportunity to verify the interpretation of the XRD data by studying the synthesised 3D-graphane by inelastic neutron scattering (INS).

We determined the g(E) spectra of powder samples of graphite hydride and deuteride by INS using the SEQUOIA neutron spectrometer at the Oak Ridge National Laboratory. The vibrational spectra of these compounds were also modelled using DFT calculations in the LDA approximation. In both the experimental and calculated vibrational spectra, there was a reliably distinguishable gap about 10 meV wide separating the acoustic band from the optical ones.

Based on the g(E) spectra obtained from the INS measurements and ab initio calculations, we calculated the temperature dependences of the lattice heat capacity Cv(T) for graphane II phases of graphite hydride and deuteride. To assess the accuracy of calculating these dependences, calorimetric measurements of the heat capacity Cp(T) of graphite hydride and deuteride at atmospheric pressure and temperatures of 130–670 K were carried out using a Perkin-Elmer DSC77 differential scanning calorimeter. Within the experimental error of 5%, the temperature dependences of the measured heat capacities Cp(T) did not differ from the calculated dependences Cv(T).

Thus, it has been proven that graphite and hydrogen compressed to high-pressures form a three-dimensional graphane II compound, and the achieved accuracy of measuring and calculating the g(E) spectrum of this compound is sufficient to accurately determine the temperature dependence of the heat capacity. Note that the g(E) spectra applicable for calculating the heat capacity have been determined so far only for two hydrides, AlH3 and MgH2 [3].

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Raman spectroscopy under megabar pressures and high magnetic fields

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Raman spectroscopy is a well-known technique for probing lattice dynamics, as well as magnetic excitations and electronic excitations such as superconducting gaps [1]. In the past few years, we upgraded the Raman facility at the High Field Magnet Laboratory (Nijmegen, NL) to enable the detection of low energy excitations (down to 0.6meV) in matter, at low temperatures (down to 1.4K) and in high magnetic fields (up to 30T and 38T) [2]. Recently, we achieved a new milestone by adding the possibility to perform Raman spectroscopy in Diamond Anvil Cells (DACs) at megabar pressures up to 230 GPa in high magnetic fields. To illustrate the scope and potential of this unique Raman experiment, a recent study on the hydride superconductor compounds H3S and YHx will be presented.

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Lifshitz transition at the onset of superconductivity in TiSe2

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Superconductivity can be driven by density-wave fluctuations in prominent unconventional superconductors. The emergent superconductivity typically exists in a dome around the quantum critical point (QCP) where the density-wave order is suppressed to zero temperature. Here, the fluctuations are strongest and hence drive superconductivity. So far, this scheme has been observed in spin-density-wave ordered materials like CePd2Si2, iron-pnictides, organic and potentially cuprate superconductors [1–4]. It remains an open question whether charge-density-wave (CDW) fluctuations can also promote superconductivity.

CDWs form a periodic modulation of the charge with a wave vector Q and the new periodicity can lead to and be driven by electronic structure changes. Here, we present high-pressure studies of the prototypical CDW superconductor TiSe2. We demonstrate that superconductivity indeed exists in a dome around the QCP of the CDW order. Our experiments realise highest-quality hydrostatic conditions which we show to be crucial to resolve the delicate physics in TiSe2.

We reveal the full evolution of the electronic structure throughout and beyond the CDW in TiSe2 [5]. We employ high-pressure quantum oscillation studies to trace the evolution of the Fermi surface. We compute the electronic structure with DFT and a tight-binding model including the CDW order. We find excellent agreement between experiment and theory and identify key reconstructions of the Fermi surface tied to the onset of superconductivity.

The presence of electron and hole pockets connected by Q are found to be a prerequisite for superconductivity. A Lifshitz transition is unambiguously revealed at 2GPa – less than half the pressure required to reach the QCP – exactly where superconductivity emerges. We show that this Lifshitz transition gives rise to electron and hole pockets at Gamma and L. The CDW fluctuations at Q connect exactly these pockets. Our findings strongly suggest that the superconductivity in TiSe2 is driven by the CDW fluctuations between the electron and hole pockets. Most likely, the superconductivity is unconventional with a sign change between the pockets similar to earlier observations in iron pnictides. Hence, our study sheds new light on the mechanisms for unconventional superconductivity.

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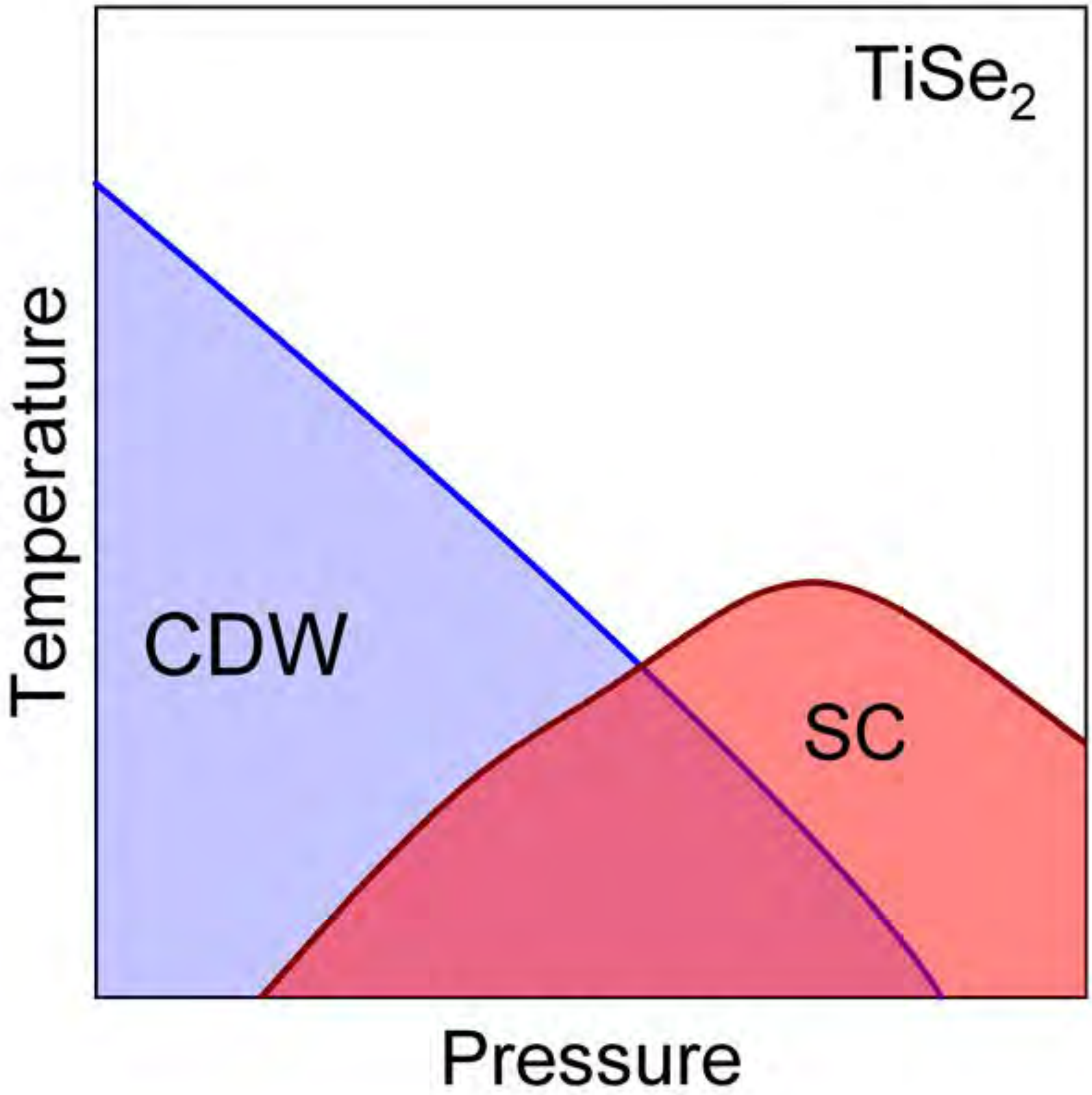
TiSe$_2$
Temperature
CDW
SC
Pressure

Clean-limit superconductivity in Hydrogen Sulphide H3S

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The discovery of superconductivity in H3S at temperatures up to 203K has started the new field of hydride high-temperature superconductivity [1]. Further superconductors with transition temperatures up to 264 K have been reported in LaH10, YH9, and other hydrides [2,3]. Synthesis and experimental studies of hydride superconductors remain challenging due to the high-pressures required. Further experimental studies will require clean samples to identify the intrinsic characteristics of the high-Tc superconductivity. However, most of the hydride superconductors cannot be obtained phase-pure due to competing stable stoichiometries and the non-selective synthesis.

We present detailed high-field studies of H3S which demonstrate that H3S exhibits clean-limit superconductivity [4]. We observe superconductivity in our resistance measurements and trace the transition in high magnetic fields. From the critical field, we extract the coherence length in agreement with earlier results [5]. Comparison with the mean free path extracted from the normal state resistance provides evidence for clean-limit superconductivity. Further evidence arises from the comparison with other high-field studies of H3S [1,5]. We present comparison with band structure calculations that demonstrate the effect of electron-phonon coupling on the Fermi velocity.

We show that fluctuation effects are in good agreement with the observed rounding of the resistive transition. We discuss the role of fluctuations in three-dimensional superconductors and compare this with commonly known two-dimensional high-Tc superconductors.

In summary, our results do not only underpin the earlier identification of superconductivity in H3S but also promote H3S as the prime candidate to study hydride high-Tc superconductivity in a clean system. Our synthesis from ammonia borane and sulphur simplifies the sample preparation compared to pure hydrogen which requires gas loading. Hence, we expect that this route will enable more researchers to study high-temperature superconductivity in hydrides.

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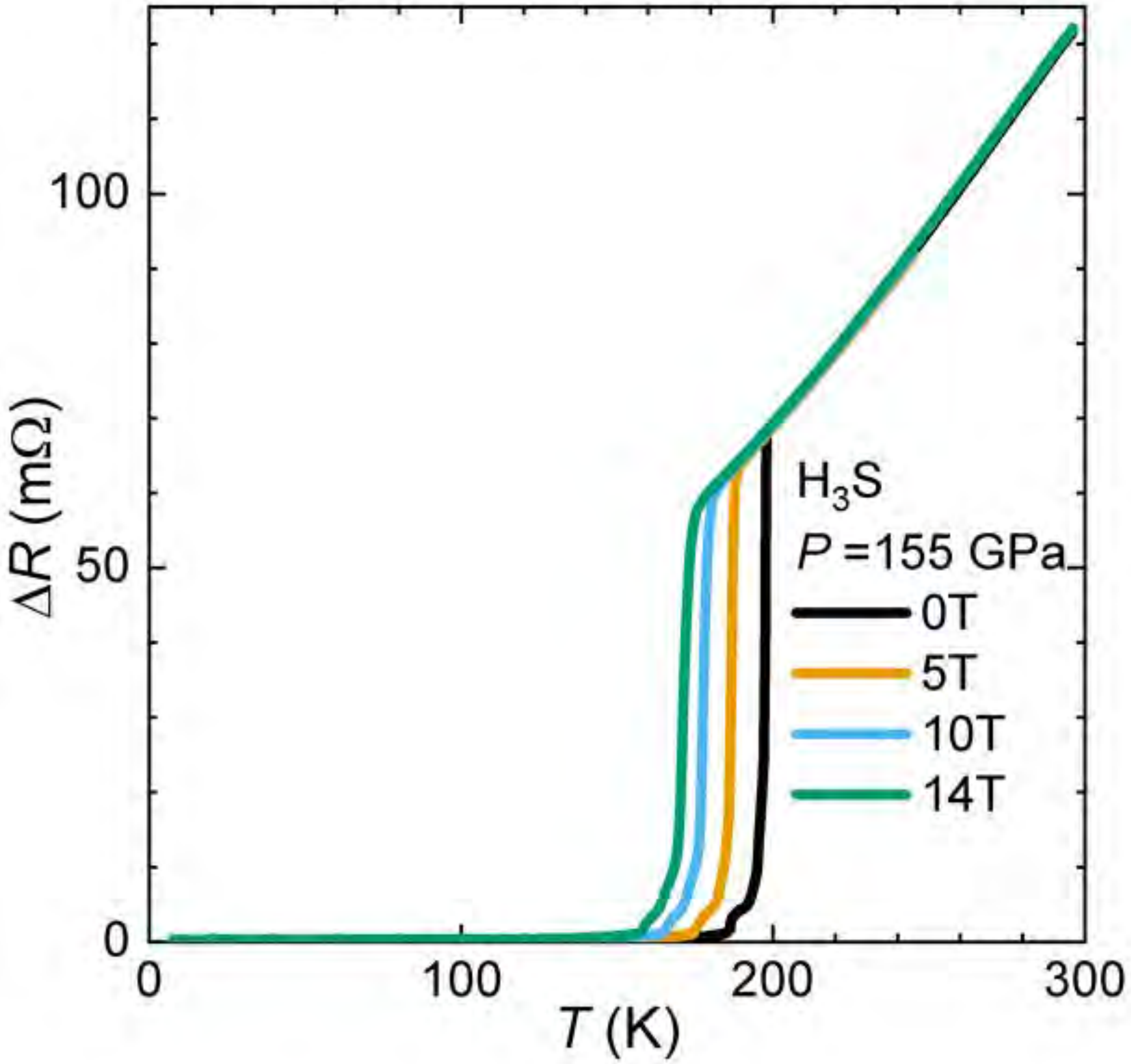
ΔR (mΩ)
100
50
0
0
100
200
300
T (K)
H_3S
P =155 GPa
0T
5T
10T
14T

Pressure-induced sorption in Cd(oba)(azpy) metal-organic framework in liquid and gaseous environment

Aleksandra Półrolniczak[1], Szymon Sobczak[1], Andrzej Katrusiak[1]
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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The study of materials capable of addressing environmental issues such as global warming, air and water pollution, and climate change is of utmost importance. A cage-like construction of metal-organic frameworks (MOFs) resulting in high porosity and high surface area combined with the variety of inorganic and organic components, makes them appropriate for gases storage or high-capacity adsorbents.

In this study, we design, synthesise and investigate a sorption properties of a highly porous MOF: Cd(oba)(azpy), where H_2oba=4,4'-oxybis(benzoic acid), azpy= 4,4'-azopyridine, in liquid and gaseous environment (Fig. 1a). The ability for absorbing of liquids were tested in diamond-anvil cell, in the environment of a small-molecule pressure transmitting medium (PTM) such as methanol, ethanol, acetonitrile, or chloroform, and in a large-molecule liquid (Daphne 7373). The compression to relatively low pressure in small-molecule PTMs triggered the transport of guests to the pores leading to its immediate amorphization (Fig. 1b) while the submersion in the non-penetrating medium preserved this material in at least 1 GPa (Fig. 1c). The gas sorption were investigated by using the self-made, high-pressure capillary device, designed to work at gas pressure from 1 to 200 atmospheres. In the two sets of the experiments conducted in CO_2 or N_2 environment, the single-crystalline Cd(oba)(azpy) demonstrated two opposite behaviours: in CO_2 quickly becoming amorphous, and N_2 showing anomalous elastic deformation preserving the crystallinity. Our results demonstrate the successful implementation of the newly designed high-pressure capillary and static compression in DAC, providing a unique insight into the elastic properties of MOFs, and a shearing new light on complex relation between a framework, and a guest impacting pressure-induced sorption capabilities.

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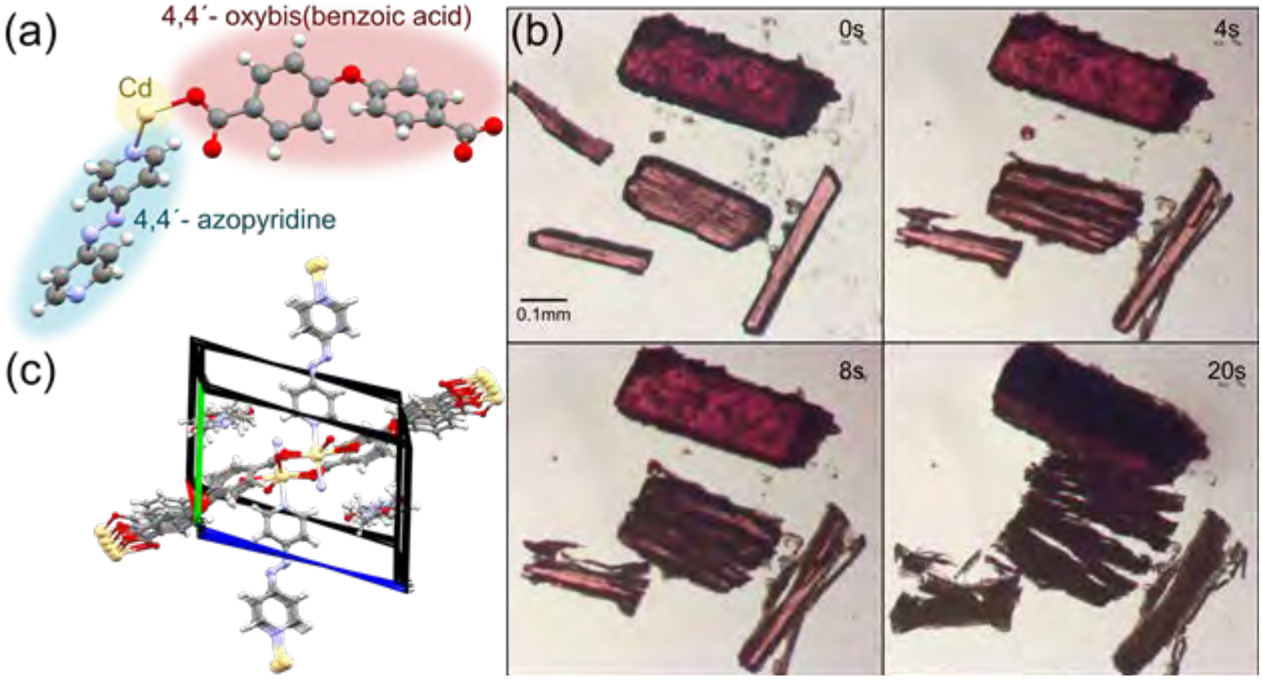
This work was supported by funding from the Polish National Science Centre: PRELUDIUM 18 No. UMO-2019/35/N/ST5/01838 and grant POWR.03.02.00-00-I026/16 co-financed by the EU European Social Fund under the Operational Program Knowledge Education Development.


(a)
4,4´- oxybis(benzoic acid)
Cd
4,4´- azopyridine
(b)
0s
4s
8s
20s
0.1mm
(c)

Development of conical diamond anvil cell for single-crystal neutron diffraction under high-pressure

Dr. Keishiro Yamashita[1], Prof. Kazuki Komatsu[1], Prof. Stefan Klotz[2], Prof. Tetsuo Irifune[3], Dr. Toru Shinmei[3], Prof. Kazumasa Sugiyama[4], Prof. Toru Kawamata[4], Prof. Hiroyuki Kagi[1]
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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Single-crystal neutron diffraction is a strong technique to obtain definitive information on the structure, especially of hydrogen-containing materials such as water ice, gas hydrate, and salt hydrates which show unique behaviours under high-pressure. Despite its advantages, in-situ observation has been limited compared to well-developed apparatus for powder samples (e.g. Paris-Edinburgh press [1]). Recently, some groups developed diamond anvils cells (DACs) for single-crystal neutron diffraction using different approaches [2–5]. Here, we developed DACs with a new concept and new materials (Figure).

In the newly designed DAC, the sample is mostly surrounded by parts made of nano-polycrystalline diamond [6], Zr-based bulk metallic glass [7], and Ti-Zr alloy. These materials are more suitable for neutron diffraction than conventional materials such as single-crystalline diamond, steel, and other alloys because of higher transmittance and less parasitic scatterings. Moreover, the sample is spatially separated from a bulky loading frame which usually limits the available neutron pathways in conventional high-pressure cells. Such materials and conical design allow the wide coverage of observable reciprocal space without angular limitation. Its feasibility was tested using two diffractometers: D9 four-circle diffractometer at ILL with monochromated neutron [8] and BL18 (SENJU) Laue-TOF diffractometer at J-PARC MLF with pulsed white neutrons [9]. The sample can be loaded both in liquid and solid forms of 0.1–0.4 mm^3 in volume. Single-crystalline specimens can be also prepared from solution under pressure [8,10] owing to the optical accessibility through the NPD anvils. The DAC still has challenges to overcome such as reachable pressure, currently up to 4.5 GPa, but the developed DAC has the potential to provide pivotally important information to elucidate the physical and chemical properties of materials.

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20 mm


10 mm
BMG back nut
Sample
NPD anvils
Gasket
BMG/TiZr cylinder

The Isotope Effect and Critical Magnetic Fields of Superconducting YH$_{6}$: A Migdal-Eliashberg Theory Approach

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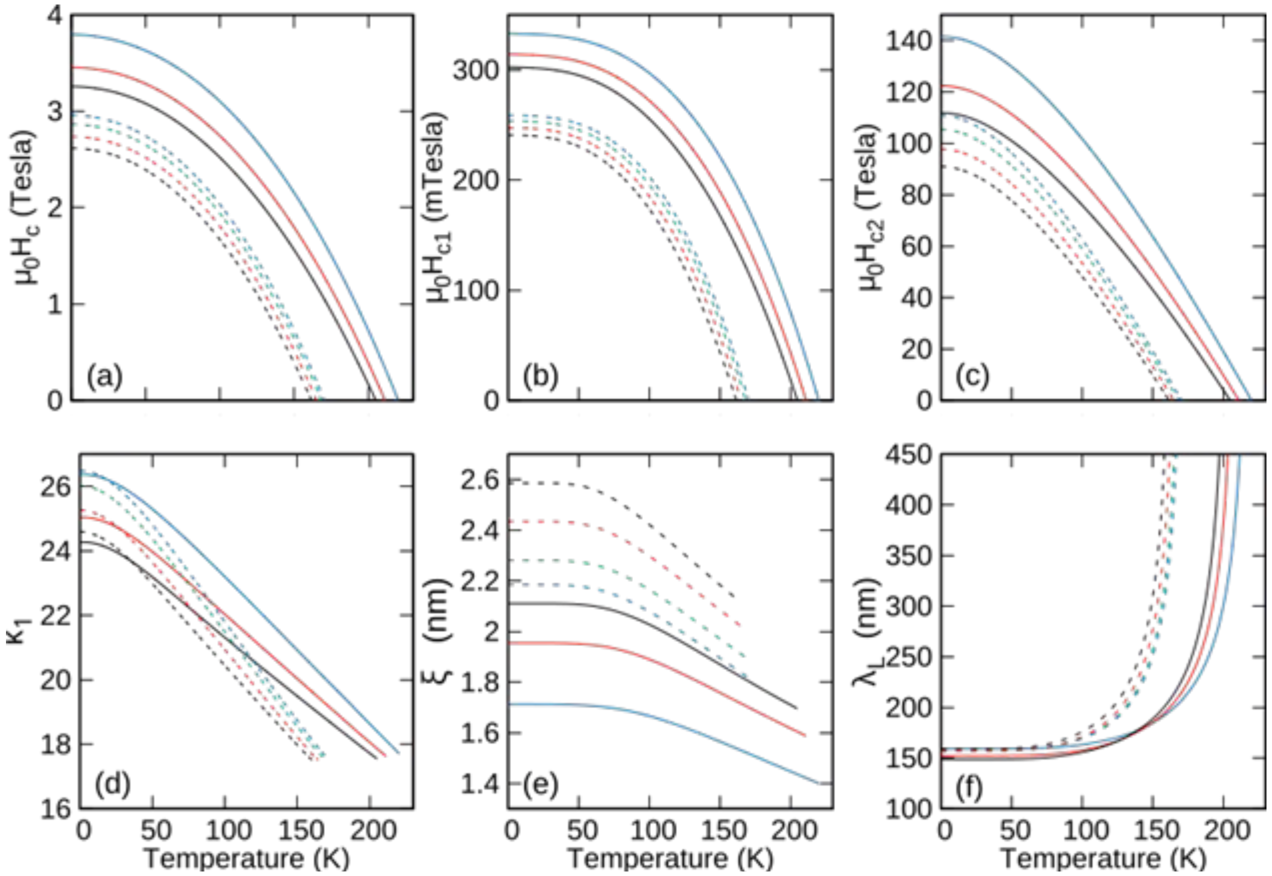
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The emergence of near-ambient temperature superconductivity under pressure in the metal hydride systems has motivated a desire to further understand such remarkable properties, specifically critical magnetic fields. YH_6 is suggested to be a departure from conventional superconductivity, due to apparent anomalous behaviour. Using density functional calculations in conjunction with Migdal-Eliashberg theory we show that in YH6 the critical temperature and the isotope effect under pressure, as well as the high critical fields, are consistent with strong-coupling conventional superconductivity; a property anticipated to extend to other related systems. Furthermore, the strong-coupling corrections occur to the expected BCS values for the Ginzburg-Landau parameter (κ_1 (T)), London penetration depth ($\lambda_{L}(T)$), electromagnetic coherence length ($\xi(T)$), and the energy gap (Δ_0).



Search for Superconductivity of Layered Iron Superhydrides Synthesised under High Temperature and High Pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Recently, various hydrides have been found to exhibit superconductivity under high-pressure (e.g. critical temperature Tc = 203 K at 150 GPa for H_3S [1]). Previous high-temperature superconductors (such as cuprate) have a layered crystal structure, while most of the hydrides with high-Tc are characterised by high symmetric crystal structure (e.g. H_3S, in which hydrogen atoms are arranged in symmetrical positions between sulfur atoms aligned in bcc). In contrast, iron superhydrides (FeH_5, FeH_6) have a layered crystal structure and are predicted to exhibit superconductivity. In addition, FeH_x have magnetic phases at lower pressure ($P < 100$ GPa) and lower hydrogen composition (x~1-4). On the other hand, superconducting phases are expected to appear at higher pressure ($P > 100$ GPa) and higher hydrogen composition (x=5, 6) [2, 3]. According to theoretical calculations [2, 3], Tc of iron hydrides FeH_5 and FeH_6 are predicted to be 51 K (130 GPa) and 43 K (150 GPa), respectively. Whereas there is a theoretical prediction that FeH_5 and FeH_6 do not exhibit superconductivity [4]. Experimentally, FeH_x (x=3, 5) have already been synthesised from the mixture of Fe and H_2 by infrared LASER heating under high-pressure [5]. In this study, we measured the temperature dependence of the electrical resistance of iron superhydrides synthesised under high temperature and high-pressure to search for superconductivity.

We used a diamond anvil cell (DAC) having the electrodes for electrical resistance measurement. Iron foil was cut into 2 µm × 2 µm × 20 µm pieces by focused-ion-beam and placed into a sample chamber in the DAC filled by ammonia borane (AB, NH_3BH_3) as a hydrogen source. After compression to 150 GPa, infrared LASER was irradiated to the iron foil to heat up and decompose AB for hydrogenation of iron. Synchrotron powder X-ray diffraction (XRD) and electrical resistance measurements were performed before, during, and after heating at BL10XU at SPring-8. Temperature dependence of the electrical resistance was measured with three terminals in the temperature range down to 150 mK using a dilution refrigerator (TRITON, OXFORD instruments).

After heating up to 2000 K and 2200 K, we obtained that XRD pattern can be explained by FeH_3 with a simple cubic unit cell and tetragonal structural FeH_5, respectively. Temperature dependence of the electrical resistance of FeH_3 and FeH_5 show that no drops indicating superconducting transition was observed.

Experiments are currently in progress to synthesise iron hydride (FeH_6) with higher hydrogen content.

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On the high-pressure phase of 1T-HfSe2

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[1]MALTA-Consolider Team and University of Oviedo, Oviedo, Spain

Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

After exhaustive searches with evolutionary structure prediction algorithms and detailed DFT optimizations, two new candidates with orthorhombic and monoclinic symmetries are proposed for the first pressure-induced polymorph of 1T-HfSe2. Previously reported hexagonal and monoclinic structures are critically examined regarding the observations reported of XRD data, equations of state, coexistence with the low-pressure trigonal polymorph in wide pressure ranges, and the pressure evolution of Raman frequency shifts. The experimental information points towards a change in the 2D dimensionality of the low-pressure laminar 1T-HfSe2 polymorph toward a so-called 3D polymeric structure. Whereas this "dimensional switching" is not accounted for by the previously proposed structures, our orthorhombic candidate shows consistency with the experimental data reported so far and constitutes a convincing alternative for the pressure-induced phase observed after 1T-HfSe2. This finding is important to understand and explain the emergence of metallicity and superconductivity in transition metal dichalcogenides as pressure is applied.

Financial support from Spanish PID2021-122585NB-C21-2 AEI project and Principado de Asturias-FEDER under project AYUD/2021/51036 is gratefully acknowledged.

High-pressure/high-temperature phase diagram of BaH_2 and the formation of barium polyhydride

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

High-temperature phase of BaH_2 (HT, Ni_2In-type structure, $P6_3/mmc$) exhibits a superionic conductivity of 0.2 S cm^{-1} at 630 °C with hydride as a carrier [1]. However, the stable condition of HT is limited to the temperature range of ~500 to 675 °C, and, therefore, it is difficult to investigate the properties over a wide temperature range. The ambient-pressure (AP) phase has been reported to undergo a pressure-induced transformation to the first high-pressure phase (HP1) at 2.5 GPa [2], and its crystal structure has been analysed to be $P6_3/mmc$, which is the same with HT, by neutron diffraction measurements [3]. This means that the stable temperature of this superionic conduction phase is extended under high-pressure (HP). In fact, HP1 has been reported to exhibit high ionic conductivity [4]. In this study, high-pressure/high-temperature (HP/HT) X-ray diffraction (XRD) measurements were performed up to about 70 GPa and 550 °C using external heating diamond-anvil-cells (DACs) to confirm the phase relationship of BaH_2. We also investigated the crystal structure of the second high-pressure phase (HP2), which appeared at ~50 GPa, by HP XRD experiments and a DFT calculation.

A commercially available BaH2 powder (99.5 %, Mitsuwa Chemicals Co., Ltd.) was loaded into DACs. HP experiments at room temperature were conducted using He or H_2 gas as pressure media. HP/HT experiments were conducted without a pressure medium or with a He medium. The pressure was determined from the shift of the fluorescence lines of ruby or Sm^2+ doped SrB_4O_7, and the temperature was measured using K-type thermocouples. The XRD experiments were performed at BL-18C and AR-NE1A of KEK-PF. Changes in the XRD pattern were observed at various pressure/temperature paths to determine the phase boundaries.

The HP/HT phase diagram of BaH_2 obtained is shown in Fig. 1. This suggests that HP1 and HT are the same phase, that is, a high-pressure high-temperature phase (HPHT). The pressure-induced phase transition from AP to HP1 is sluggish at lower temperature and the two phases coexist in the pressure range of 1.3–5.0 GPa at room temperature. The phase transition from HP1 to HP2 was observed at ~50 GPa and room temperature by XRD experiments using He medium. The DFT calculation for HP2 predicted that its crystal structure would be the AlB_2-type, similar to the previous powder XRD experiment [2]. However, the XRD pattern of BaH_2 pressurised in hydrogen showed many diffraction peaks, which did not appear in the pattern in He and without a medium. This suggests the formation of a new Ba-H polyhydride. These diffraction peaks did not correspond to the structures such as BaH6 predicted by the calculations of Hooper et al. [5].

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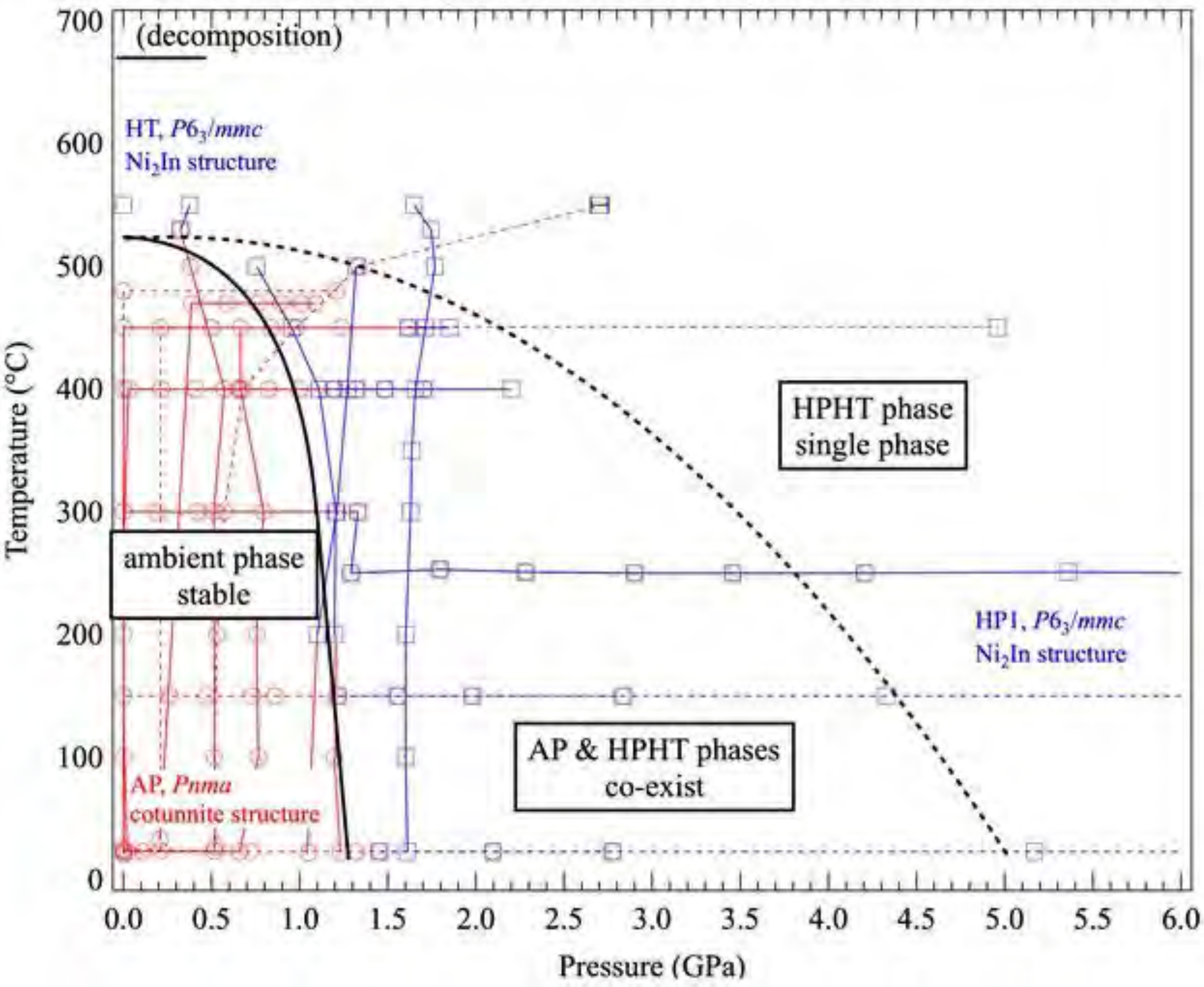

Fig. 1. Phase diagram of BaH_2 obtained by high-pressure/high-temperature X-ray diffraction measurements. A red circle and a blue square represent the ambient phase and the high-pressure phase (the high-pressure/high-temperature phase), respectively.

Optical properties of SiV and GeV colour centres in nanodiamonds under hydrostatic pressures up to 180 GPa

Marie-Pierre Adam[1], Baptiste Vindolet[1], Loïc Toraille[1,2], Mayeul Chipaux[3], Antoine Hilberer[1], Géraud Dupuy[1], Lukas Razinkovas[4], Audrius Alkauskas[4], Gergo Thiering[5], Adam Gali[5,6], Mary De Feudis[7], Midrel Wilfried Ngandeu Ngambou[7], Jocelyn Achard[7], Alexandre Tallaire[7], Martin Schmidt[1], Christoph Becher[3], Jean-François Roch[1]

[1]Lumin, Université Paris-Saclay, CNRS, ENS Paris-Saclay, CentraleSupelec, Gif-sur-Yvette, France, [2]CEA, DAM, Bruyères-le-Châtel, France, [3]Fachrichtung Physik, Universität des Saarlandes, Saarbrücken, Germany, [4]Center for Physical Sciences and Technology, Vilnius, Lithuania, [5]Institute for Solid State Physics and Optics, Wigner Research Centre for Physics, Budapest, Hungary, [6]Department of Atomic Physics, Institute of Physics, Budapest University of Technology and Economics, Budapest, Hungary, [7]Laboratoire des Sciences des Procédés et des Matériaux, Villetaneuse, France

Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Diamond anvil cell (DAC) technology is widely used to study materials under high-pressure. These materials can exhibit exotic phases of matter such as superconductivity with exceptionally high critical temperatures. The main difficulty with using the DAC lies in the challenge to measure magnetic properties at high-pressure (above 100 GPa) due to the minute sample size. Using diamond quantum defect, such as nitrogen-vacancy (NV) centre, as local magnetic probes is an effective alternative approach that allows Meissner effect detection [1]. This method has been implemented up to 130 GPa [2]. However, the use of the NV centre as a high-pressure magnetic sensor requires the implementation of the microwave excitation compatible with the constraints associated with the DAC. It can also suffer from the detrimental influence of off-axis magnetic field that may prevent its practical use at high magnetic field. Alternative diamond point defects that have attracted considerable attention in the past decade are group-IV-vacancy centres (G4V). G4V centres offer the option of all-optical, microwave-free, coherent control of their spin states.

Here we report the measurement of the optical properties of silicon-vacancy (SiV) and germanium-vacancy (GeV) colour centres in nanodiamonds under hydrostatic pressure up to 180 GPa [3]. The nanodiamonds were synthesised by Si- or Ge-doped plasma-assisted chemical vapor deposition and, for our experiment, pressurised in a DAC. Under hydrostatic pressure, we observe blueshifts of the SiV and GeV zero-phonon lines by 17 THz (70 meV) and 78 THz (320 meV), respectively. These measured pressure-induced shifts are in good agreement with ab initio calculations that take into account the lattice compression based on the equation of state of diamond. These calculations were extended to the case of the tin-vacancy (SnV) centre. Our results provide guidance on the use of G4V centres as quantum sensors under extreme pressures that will exploit their specific optical and spin properties induced by their intrinsic inversion-symmetric structure.

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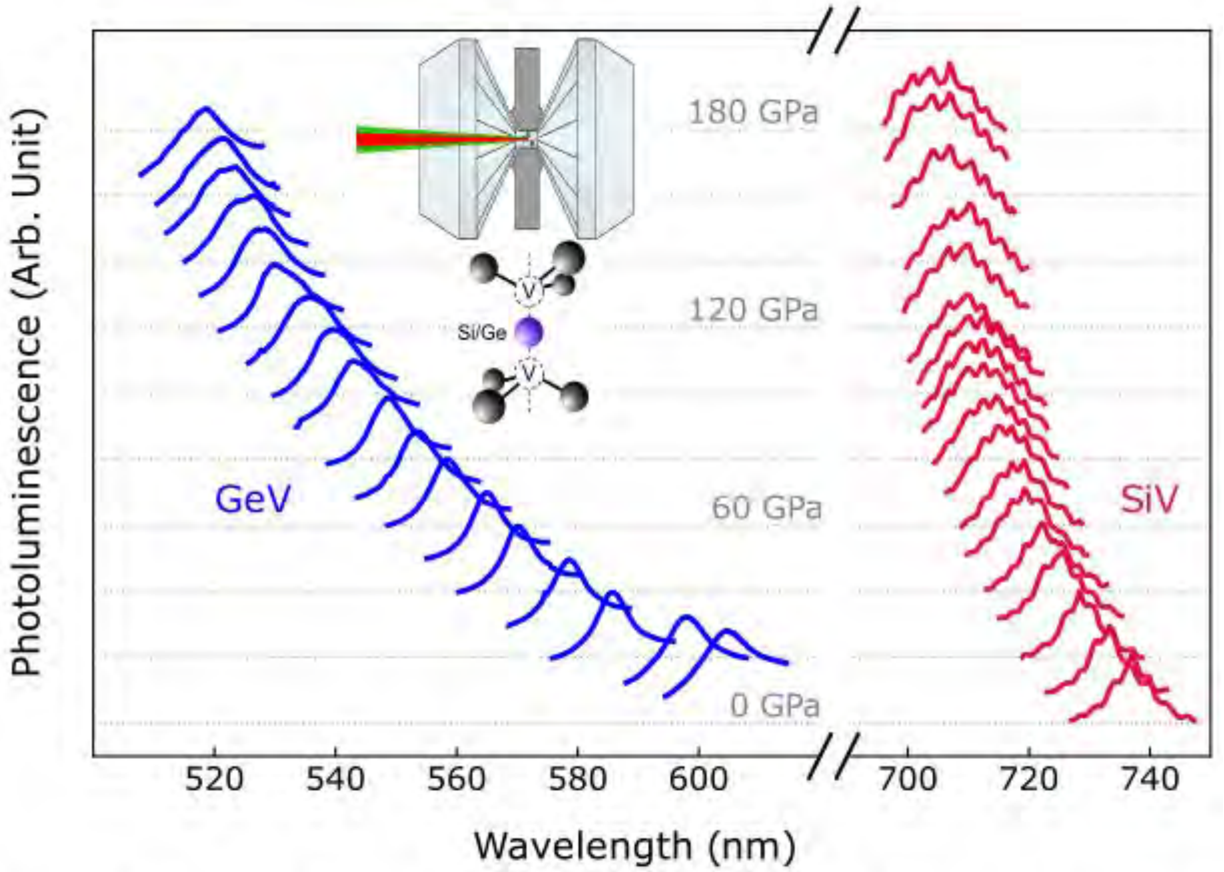

Figure 1: Pressure dependence of the photoluminescence spectra of SiV centers (in red, right) and GeV centers (in blue, left) in CVD-grown nanodiamonds. Inset: Scheme of the DAC loaded with the Si- or Ge-dopped nanodiamonds.

Transmission electron microstructure studies of Ti40Zr20Nb20Hf5Ta15 high entropy alloy after laser-heated diamond anvil cell experiments

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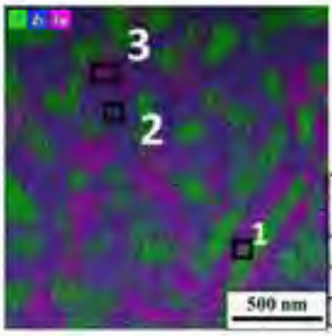
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Laser Heated Diamond Anvil Cell (LH-DAC) with in situ synchrotron analysis was used to study the effect of melting at nitrogen atmosphere under ultra-pressure of Ti40Zr20Nb20Hf5Ta15 (%at) high entropy alloy. The reaction of elements compounds with an excess of nitrogen loaded in diamond-anvil cells (DAC), and applying laser heating (2500 K), where the melted sample is next rapidly cooled, allows the formation of new non-equilibrium compounds. Rietveld refinement of patterns registered at 32 GPa, after heating, confirms the presence of complex nitrogen contains phases: M_2N_3, (Pnma), M_3N_5, (Pmnb), M_4N_5 (I4/m), where M = Ti, Zr, Nb, Hf, Ta. Furthermore, the release of pressure revealed that the nitrides in the supersaturated alloy were stable even at ambient pressure. Transmission Electron Microscopy (TEM) studies were conducted to describe the post-processing evolution of the microstructure. For this purpose, a special procedure of TEM thin foil preparation was developed using a Focus Iron Beam (FIB) from hydrostatically compressed 20 µm grains.
A High Angle Annular Dark-Field imaging (HAADF) with mapping of element distribution is shown in Fig. 1. Nano-size lamellae morphology of a structure with segregation of elements are clearly visible, suggesting preferential phase formation under the non-equilibrium condition of crystallisation. Three groups of phases with dominant elements containing nitrogen have been identified: Ti-, Zr-, and NbTa. A large number of structural defects were observed in the bright field image. Selected area electron diffraction patterns confirm the presence of Ta_2N_3 with space group Pnam.

Acknowledgement:
This research was supported by Polish science financial resources The National Science Centre, Poland, project No. 2021/41/B/ST8/03758 entitled: Development of new high entropy nitrides composites synthesised at high-pressure and temperature.



Place	Element [%at.]					
	N	Ti	Zr	Nb	Hf	Ta
1	31.0	55.9	1.5	7.8	0.7	3.1
2	4.2	19.6	37.8	14.9	9.4	14.1
3	15.8	5.3	8.6	35.1	3.7	31.5

Figure 1 HAADF images of Ti40Zr20Nb20Hf5Ta15 at.% hydrostatically compressed at 32 GPa of nitrogen at melting temperature and pressure release.

Application of Quantum Diamond Magnetometry to High-Tc Cuprate Superconductivity

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Quantum diamond magnetometry based on the spin properties of nitrogen-vacancy (NV) centres is a well-established technique to measure magnetic fields with a unique combination of resolution and sensitivity [1]. This sensing method can be applied to quantitatively measure the magnetic properties of a sample of micrometre size pressurised in a diamond anvil cell (DAC) [2]. By mapping the magnetic field distribution around a sample, this method provides a direct detection of the Meissner effect and can be used to determine the pressure dependence of the superconducting critical temperature.

Here we report the application of this method to detect the superconducting properties of a doped mercury based cuprate $Hg_2Ba_2Ca_2Cu_3O_{10}$ (Hg-1223) under pressures up to 30 GPa. We observe a good agreement with the dome-shape pressure dependence of the superconducting critical temperature [3]. By tailoring the anvil tip to ensure controlled quasi-hydrostatic environment for the NV sensors [4], this technique can be extended to an unexplored pressure range where a predicted pressure-induced change of the band structure could induce a strong increase of the critical temperature, as observed in $Bi_2Sr_2Ca_2Cu_3O_{10}$ (Bi-2223) [5].

We will also report the implementation of wide-field quantum diamond magnetometry to spatially resolve the Meissner effect. This technique reveals the existence at micrometre length scale of sample inhomogeneities that induce local modification of the critical temperature. This spatial information is inaccessible to most other techniques that probe the properties of the sample as a whole.

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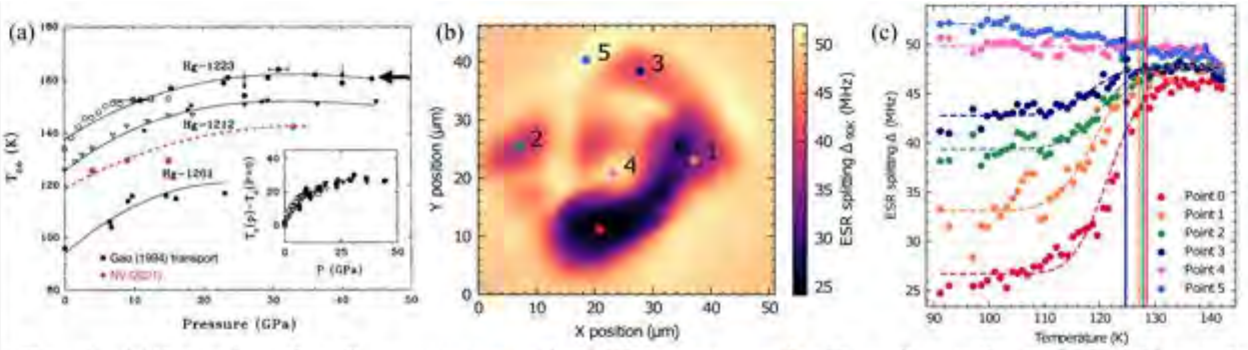

Figure 1 – (a) Comparison of our Tc measurements on the Hg1223 cuprate sample with previous studies. (b) Wide-field map of the optically detected Electron Spin Resonance (ESR) splitting directly proportional to magnetic field over the Hg1223 cuprate sample at 3 GPa. (c) Variation of the ESR splitting with temperature on 5 different points of the sample at 3 GPa. The fitted Tc are indicated by the corresponding-colored vertical lines.

Sound velocity, second shock velocity and off-Hugoniot measurements of lead compressed up to 83 GPa

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Two series of shock wave experiments to investigate the sound velocity and second shock velocity of lead were carried out using the powder gun facility at Soreq NRC. An overtaking method [1] has been utilised in both types of experiments. A Photon Doppler velocimeter (PDV) was used to monitor the sample/window (either PMMA or LiF) interface velocity. The waveforms recorded in sound speed and re-shock experiments exhibit a shock wave front followed by a plateau and eventually by a release wave or a second shock wave front, respectively. Values of sound velocity obtained in present study are in good agreement with literature data for lead. Off-Hugoniot state of lead behind the release wave emanating from the sample/window interface were estimated as well. Mie-Gruneisen equation of state was calibrated to the data presented in this work.

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Ni2+ broadband infrared emission in varying temperature and pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Phosphor-converted infrared light-emitting diodes (pc-LEDs) are an emerging alternative to conventional InGaAs and GaAs-based semiconductor LEDs, and traditional infrared light sources such as tungsten halogen lamps and laser diodes, possessing good versatility and temperature characteristics. The broadband near-infrared (NIR) pc-LED device based on blue LED pumped Cr3+ activated phosphors is known for the NIR emission ranges of 600–1100 nm. Cr3+ activated phosphors with exceptional broadband NIR luminescence properties are reported through modulating the crystal field strength, but the maximum emission wavelength is less than 1000 nm. The second overtone and vibrational combinations of basic organic bonds in spectroscopy absorb shortwave infrared (SWR) light more effectively than NIR. Hence, a pc-LED light source with broadband SWIR emissions in 900–1700 nm ranges is desperately needed for more exciting solutions in spectroscopy, optical coherence tomography, optical communication, and noninvasive medical imaging applications.

Here are presented examples of such Ni2+ infrared broadband emission induced by the Cr3+ emission. Results consist of the photoluminescence spectroscopy complemented by photoluminescence excitation and time - resolved spectroscopy in varying temperature as well as high-pressure. Shared qualities of both Ni2+ and Cr3+ are presented here, and the explanation for the luminescence and its association with the forming of pairs or so-called clusters is provided.

Chemical and mechanical pressure influence on luminescence properties of near-infrared phosphors

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Investigating the optical properties of inorganic compounds doped with transition metals is essential in high-pressure studies using the diamond anvil cell. By exposing the phosphor material to high-pressure, we directly influence the interaction of the crystal environment to luminescent centres and cause significant changes in the studied systems' energy structures. The overall conclusion from the structural point of view is the same effect that mechanical pressure can also be observed when chemical pressure is applied. Namely, incorporating the ion with a bigger ionic radius in the crystal lattice causes an increase in the atomic distance in the crystal, decreasing the crystal field strength around the ion. Opposite, incorporating the ion with a smaller ionic radius causes a decrease in the atomic distance in the crystal lattice, increasing the crystal field strength.

In this study, we aim to compare the changes in the luminescence for two reference series of samples: Ga2AlxO3:0.02Cr3+ (GAOC) and Ga2-xScxO3:Cr3+ (GSOC). We found that GAOC has qualitatively the same, while GSOC has different behaviour in the chemical and mechanical pressure dependences of the luminescence properties. In the case of Al, we can consider it as ion substitution in the neighbour unit cell, which changes the crystal properties like mechanical pressure dose. When Ga2O3 is co-doped with the Sc ions, it causes lattice distortion in the Cr3+ local environment, and by applying pressure for GSOC, we reduce the volume of the cell, but we cannot reverse the distortion caused by the co-doping.

Visible to mid-IR reflectivity of materials under extreme pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

A new experimental set up aiming at studying the optical response, extending from the visible to the mid-infrared, of materials under pressure has been designed. The set up allows both transmission and reflection geometry for such analysis. It combines Fourier transform infrared (FTIR) and visible spectroscopy with wide-band lasers and classical light sources to provide quantitative information over a broad and continuous spectral range from 400 nm to 10 µm (25000 to 1000 cm^-1). This is compatible with sample sizes down to a few microns at megabar pressure inside diamond anvil cells without using synchrotron light sources.

We report the optical reflectivity measurements obtained on small metallic particles and on hydrides, as a function of pressure. Such approach can be used to understand the electronic and lattice properties of materials and their modification under pressure, in various systems from semiconductors to metals and superconductors. Optical reflectivity is one of the hallmarks for studying, besides vibrational modes, electronic bands shifting, collective excitations like plasmon resonances, or gap closures or openings during insulator-metal transitions.

The capabilities of our setup will be illustrated on silver, aluminium and gold micron-sized particles, as well as on hydride AlH3 under pressure. The accuracy and reproducibility of the reflectivity measurements require control over several parameters which will be discussed.

High-pressure magnetic measurements of β-phase UH3

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Uranium and hydrogen readily react to form uranium hydride (UH3). Despite being discovered as the first 5f ferromagnet in 1952, there are still some properties of UH3 that are not well understood. The magnetism in 5f materials is described by the Hill limit. The Hill limit is an empirical boundary between magnetic and non-magnetic actinide materials, with the magnetic materials existing above this specified limit. The limit for uranium is given as do = 3.4 A – 3.5 Å [1]. The αUH3-phase sits above the limit with U-U interatomic distances of 3.6 Å [2], while the βUH3-phase falls below, with U-U interatomic distances of 3.31 Å [3]. Therefore, it is expected that only the α-phase will have a ferromagnetic state. Remarkably, both phases exhibit ferromagnetism and share a curie temperature of around 175 K, despite having vastly different U-U bond lengths [4]. In the literature, there is a remarkable paucity of information around how the ferromagnetism arises in the material and how the magnetic properties change under pressure.

A previous study conducted by Andreev et al. [5] has investigated the pressure dependence of the magnetic properties seen in β-phase UH3 up to pressures of 1 GPa. The study presented data which showed an inverse relationship between curie temperature (Tc)/magnetic moment (M) and pressure. Therefore, it will be of interest to continue these studies and investigate how the ferromagnetic state changes as pressure is increased. It has been possible to estimate the critical pressure at which the curie temperature becomes 0 K for β-phase UH3. This was achieved by using a correlation provided by Andreev et al. and was calculated to be around 6.1 GPa [5].

The work conducted at the University of Bristol will aim to study the magnetic properties of β-phase UH3 up-to and beyond 6 GPa. Our investigation will probe for potentially novel magnetic and electronic phases, such as a superconducting phase that may arise at the critical pressure. Hall magnetometry will be utilised in the proposed work to measure the magnetization of β-phase UH3 under pressure. The high-pressures required for this research will be conducted in a diamond anvil cell (DAC). The small culet diameter of the diamonds range from between 800 µm – 100 µm in diameter, these diameters allow for the creation of high-pressure environments ranging from 15 GPa – 75 GPa respectively. In order to carry out Hall magnetometry under pressure, we will employ thin film devices on the diamond anvil. We will present details regarding the fabrication and characterization of the hall sensor.

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Phase diagram of ethane to 450 K and 120 GPa

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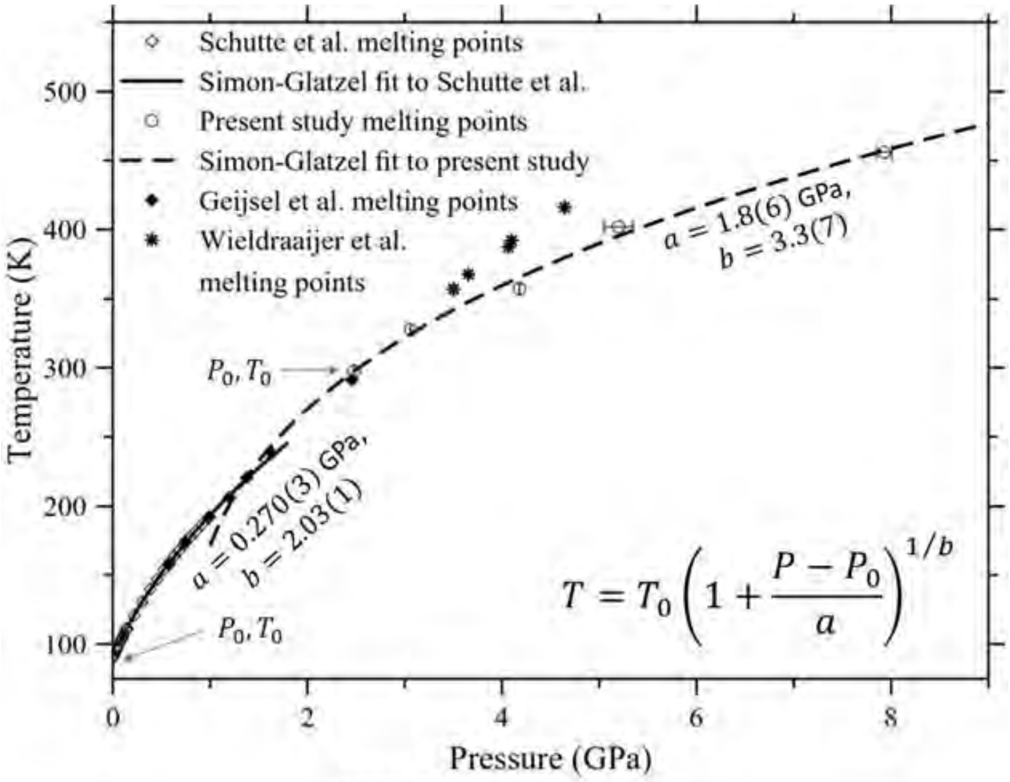
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

We have conducted a series of Raman scattering experiments on liquid, supercritical fluid and solid ethane (C_2H_6) in the diamond anvil cell. This has enabled us to map out the melting curve to 450 K. We have noticed that, catalysed by ruby or Sm:YAG pressure markers, the ethane molecule can decompose above the surprisingly low temperature of 375 K.

Measurement of melting above 450 K using the diamond anvil Raman pressure gauge indicated that a temperature correction to this pressure gauge may be required. At 300 K, on the other hand, no evidence for decomposition of the ethane molecule was observed up to 120 GPa, the highest pressure reached in our work.

In the liquid state we observe the Frenkel line at 300 K (one of only two examples to our knowledge where it has been directly observed in the subcritical region) but are unable to confirm its presence of otherwise above 300 K due to the higher errors in pressure measurement.

As far as solid phases are concerned, our findings are in agreement with those studies that suggest freezing at 300 K is into phase IV (rather than phase II), and that there are potentially transitions both at 300 K and above 300 K to phases for which full structural characterization is still required.



Ethane melting curve fits

J.E. Proctor, J.E. Spender and H.T. Gould, J. Phys. Chem. C **126**, 10792 (2022)

Raman scattering study of liquid and solid propane to 60 GPa at 300 K

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

We have performed Raman spectroscopy experiments on liquid and solid propane (C_3H_8) to 60 GPa at 300 K. Our observations in the solid state are similar to those of previous studies. We do, however, observe the solid-liquid transition using both Raman spectroscopy and visual observation at significantly lower pressure (2.7 GPa) than previously reported (3.2 GPa). In the liquid state we did not detect any sign of the Frenkel line despite collecting data at extremely low pressure (5 data points beneath 0.5 GPa). 300 K is a significantly subcritical temperature for propane (the critical temperature is 370 K) so these findings tentatively support the hypothesis that the Frenkel line (at least as defined according to the Raman scattering) terminates on the vapour pressure curve at ca. 0.8T_C.

Modelling of liquid internal energy and heat capacity over a wide pressure-temperature range from first principles

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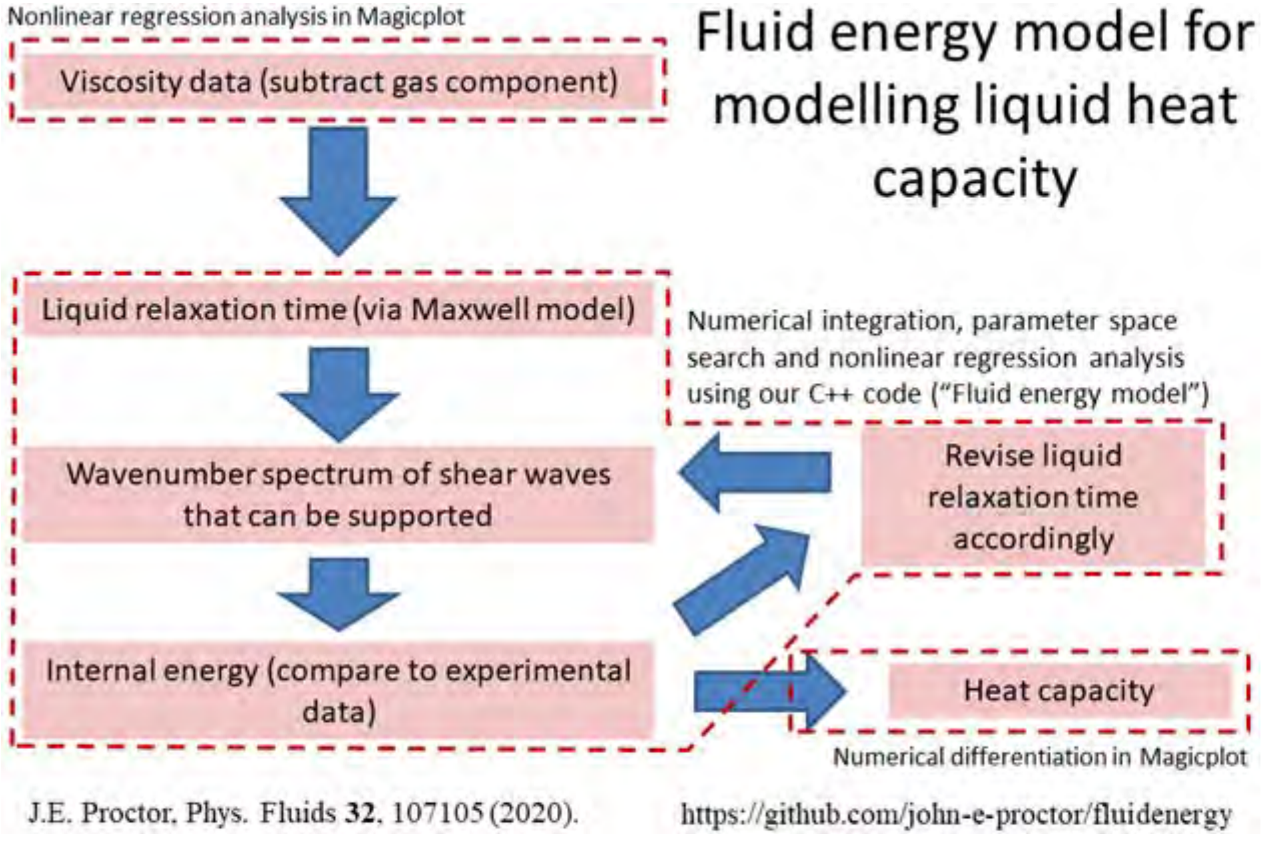
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Recently, there have been significant theoretical advances in our understanding of liquids and dense supercritical fluids based on their ability to support high frequency transverse (shear) waves. Here, we have constructed a new computer model using these recent theoretical findings (the phonon theory of liquid thermodynamics) to model liquid internal energy across a wide pressure–temperature range. We have applied it to a number of real liquids in both the subcritical regime and the supercritical regime, in which the liquid state is demarcated by the Frenkel line. Our fitting to experimental data in a wide pressure–temperature range has allowed us to test the new theoretical model with hitherto unprecedented rigor. We have quantified the degree to which the prediction of internal energy and heat capacity is constrained by the different input parameters: the liquid relaxation time (initially obtained from the viscosity), the Debye wavenumber, and the infinite-frequency shear modulus. The model is successfully applied to output the internal energy and heat capacity data for several different fluids (Ar, Ne, Kr, N_2, and CO_2) over a range of densities and temperatures.

We find that the predicted heat capacities are extremely sensitive to the values used for the liquid relaxation time. If these are calculated directly from the viscosity data, then, in some cases, changes within the margins of the experimental error in the viscosity data can cause the heat capacity to exhibit a completely different trend as a function of temperature. These findings indicate that the phonon theory of liquid thermodynamics cannot be used for a priori prediction of liquid heat capacity, but a revised implementation of the theory could be used to a priori prediction of liquid viscosity.



Fluid energy model for modelling liquid heat capacity

Nonlinear regression analysis in Magicplot

Viscosity data (subtract gas component)

Liquid relaxation time (via Maxwell model)

Wavenumber spectrum of shear waves that can be supported

Internal energy (compare to experimental data)

Numerical integration, parameter space search and nonlinear regression analysis using our C++ code ("Fluid energy model")

Revise liquid relaxation time accordingly

Heat capacity

Numerical differentiation in Magicplot

J.E. Proctor, Phys. Fluids **32**, 107105 (2020).

https://github.com/john-e-proctor/fluidenergy

High-pressure phase of Iridium-based Sr2BIr06 (B = Ca, Zn) Double Perovskites

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Iridium oxides, with the spin-orbit (SOC), crystal field (CF), electronic bandwidth, Coulomb, and exchange interactions having energy comparable scales, provide fascinating electronic and magnetic properties. According to theoretical predictions, some of them behave as different type of Mott insulators, others are Weyl semimetals, a few of them are Hall spin systems, and, for example, Sr_2CrIrO_6 might exhibit the highest TC ever reported for half-metallic ferromagnets. The family of Sr_2BIrO_6 double-perovskite-type compounds shows the highest electronic versatility: depending on the B ion the valence of iridium can be interchanged between Ir4+, Ir5+, and Ir6+ with the corresponding drastic effect that such valence states has on the Ir-O bond covalency degree in the compound. The Sr_2BIrO_6 compounds which have Ir6+ (with Mg, Ca, Zn, Ni) crystallise in a $P2_1/n$ monoclinic structure, in which Ir and B are always occupying octahedral sites. Considering the low symmetry of their structures and the fact that pressure has a direct effect on both the SOC and the CF, studying the structural behaviour of iridium oxides under compression is a requirement to understand their properties. So far, only one high-pressure work exists studying Sr_2NiIrO_6 and showing a structural phase transition around 8 GPa but leaving the structural solution open [1].
In this work, we have performed high-pressure powder XRD to follow the evolution of the crystal structure of both Sr_2CaIrO_6 and Sr_2ZnIrO_6 up to 16 GPa. Our results show the occurrence of structural phase transitions in both compounds at 6 and 8 GPa, respectively. Although the phase transitions in both compounds are isosymmetric ($P2_1/n \rightarrow P2_1/n$) each compound undergoes a phase transition to a different structure. The high-pressure phase of Sr_2ZnIrO_6 is the result of a collapse of the a-axis and an enlargement of the c-axis while the high-pressure phase of Sr_2CaIrO_6 is the result of a collapse of the c-axis. Both phase transitions involve a volume contraction of their unit-cells of ~2%. The stabilities of the high-pressure phases are confirmed by our ab initio calculations.

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Evolution of the full energy structure of Mn4+ in fluoride phosphors under high-pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The research concerns the pressure dependence of photoluminescence excitation and emission spectra of fluoride phosphors doped with Mn4+: $KNaSiF_6$:Mn4+, Rb_2GeF_6:Mn4+, Na_3HTiF_8:Mn4+. The pressure-dependent energies of $^4T_2$, $^4T_1$, and ^2E crystal field subterms of Mn4+ have been determined for these hosts in the 0-30 GPa pressure range. Strong blueshift of the $^4T_2$ and $^4T_1$ subterms was found, as expected from the Tanabe-Sugano diagram for Mn4+ (d^3). At the same time, the 2E emitting state exhibited redshift under pressure – an effect opposite to the prediction of the Tanabe-Sugano diagram. This was attributed to the pressure-driven nephelauxetic effect and clearly shows the requirement of taking into account the Racah parameters for a correct description of Mn4+ emission under pressure. The pressure dependence of crystal field strength parameter Dq and Racah parameters B and C were determined based on experimental data. Finally, the knowledge of the pressure dependence of Dq and Racah parameters allowed us to calculate the full energy structure of d 3 configuration of Mn4+ for $KNaSiF_6$:Mn4+, Rb_2GeF_6:Mn4+, Na_3HTiF_8:Mn4+ in the pressure range of 0 – 30 GPa. The calculations reproduced the redshift of the ^2E emitting state with pressure, as well as gave the pressure shift direction and magnitude for all crystal field subterms of Mn4+ up to 50 000 cm-1 (i.e. the equivalent of Tanabe-Sugano diagram for high-pressure experiments). The approach presented in this paper can be easily extended for calculating the energy structure of Cr3+ under pressure and other transition metal ions.

Impact of ionic quantum fluctuations on the thermodynamic stability and superconductivity of LaBH8

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The prediction of a meta-stable high-symmetry Fm-3m phase of LaBH8 [1,2] predicted by Di Cataldo & Liang suggests that high superconducting critical temperatures at affordable pressures is achievable through ternary hydrogen-rich compounds. The structure exhibits a superconducting critical temperature of 120 K at a pressure of 50 GPa. Furthermore, classical structure prediction calculations suggest the structure to remain dynamically stable down to 40 GPa.

In this study, we expand on the authors works by making use of first-principles calculations within density functional theory, and the stochastic self-consistent harmonic approximation [3] for the treatment of quantum anharmonic effects. The introduction of these effects has a non-negligible impact on both phase stability and superconducting properties of hydrogen rich materials. Previous studies have shown that strong renormalizations of the crystal structure and phonon spectra are to be expected for these systems [4]. The hope is then that quantum anharmonic effects could sustain the LaBH8 structure down to lower pressures.

Contrary to expectations, our results show that quantum anharmonic effects drive the system dynamically unstable below 77 GPa, a much higher pressure than the 45 GPa expected classically. Quantum anharmonic effects stretch the covalent B-H bond in the BH8 units of the structure, and consequently, soften all hydrogen-character modes. Above 77 GPa Fm-3m LaBH8 remains metastable, and interestingly, its superconducting critical temperature is largely enhanced, reaching critical temperatures around 160 K at the verge of the dynamical instability. In conclusion our results suggest that low-pressure metastable phases with covalently bonded symmetric XH8 units will be destabilised by ionic quantum fluctuations.

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Characterization of the ε'-Fe_2O_3 phase under extreme conditions

Samuel Gallego Parra[1], Juan Ángel Sans Tresserras[2], Gastón Garbarino[1], Virginia Monteseguro[3], Catalin Popescu[4], Arnau Romaguera[5], Zheng Ma[5], Martí Gich[5], Jose Luis García Muñoz[5]
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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Iron oxide (Fe_2O_3) crystallises in several polymorphs, from α (hematite) and γ (maghemite) phases broadly employed, to the less studied β and ε phases. The ε phase can be solely grown in nanoparticles, nanotubes or thin films, making it difficult to synthesise insolate [1]. As a result, the ε phase is observed as an intermediate product of transformation between other polymorphs (β, γ and α phases). This metastable phase, the ε-Fe_2O_3, exhibits a non-centrosymmetric orthorhombic structure (s.g. 33, $Pna2_1$, Z=8). Four non-equivalent Fe atoms are in two different environments: three of them in distorted FeO_6 octahedral units and the fourth in FeO_4 tetrahedral units. Such a distortion presents two of these FeO_6 octahedral units that their real coordination is closer to 5+1, more than 6, due to a longer Fe-O bond length as compared to the rest of the bonds.

By confining Fe_2O_3 nanoparticles in a silica matrix to prevent their growth while annealing above 1000ºC it is possible to obtain pure ε-Fe_2O_3 nanoparticles (powders) with very high thermal stability (in the range between 1100-1400 ºC [2,3]). On the other hand, its pressure behaviour was addressed for the first time in our previous work [4], where ε-Fe_2O_3 kept stable up to 27 GPa. Above such pressure, a volume collapse was observed of 2%, which was associated with a 1st order structural transition to a new Fe_2O_3 phase, the ε'-Fe_2O_3. Synchrotron-based angle-dispersive X-ray diffraction (ADXRD), extended X-ray absorption spectroscopy (EXAFS) and Mössbauer spectroscopy (SMS) measurements corroborated that FeO_4 tetrahedral units became 6-fold coordinated above this volume collapse. However, the crystal structure of this ε'-Fe_2O_3 could not be unambiguously solved.

In this work, we have revisited the pressure behaviour of the ε'-Fe_2O_3 by a joint experimental and theoretical approach by using high-pressure ADXRD and Raman measurements supported by ab initio calculations. Within this experimental and theoretical framework, we aim to identify the nature of the ε'-Fe_2O_3, which suits the changes observed in the coordination stressed in our earlier work.

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Combining high-pressure and low temperature with single crystal resonant X-ray diffraction, P09, PETRA III

Dr. Christian Plueckthun[1], Dr. Jennifer Sears[1], Dr. Pablo Bereciartua[1], Dr. Konstantin Glazyrin[1], Dr. Jonathan Warren[2], Dr. Maximilian Kusch[3], Dr. Marein Rahn[3], Dr. Jochen Geck[3], Dr. Sonia Francoual[1]

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Resonant X-ray diffraction is a unique and powerful technique to probe and disentangle the charge, spin and orbital degrees of freedom in quantum materials and/or understand how different magnetic species might order in magnetic alloys thanks to the element specificity. In quantum materials, changes in temperature or application of a high external magnetic field (e.g. by cryocooled magnets) modify the magnetic and electronic Hamiltonians (for instance crystal field, or superexchange terms) and might result in new states of order. Another parameter gives rise to unexpected effects in the magnetic and electronic response of those materials, namely pressure. 1 GPa pressure equals an energy density increase of 6.3 meV Å-3 and the compression of the crystal lattice modifies the bond distance and angles, thus the electronic and magnetic interactions. Combining high-pressure (HP) with low temperatures (low T) and resonant X-ray diffraction offers thus a valuable tool to understand the (P,T) phase diagram and probe the different degrees of freedom in quantum materials. With the current contribution we present the current status and recent developments of the high-pressure and low temperature diamond anvil cell setup at beamline P09, PETRA III, DESY, Germany, which enables resonant X-ray diffraction measurements up to 30 GPa pressure at temperatures between cryogenic (5 K) and 300 K. We do bring proof of concept, presenting magnetic scattering data collected at the iridium L_3 (11217 eV) absorption edge in layered canted antiferromagnet Sr_2IrO_4.

Keywords: High-pressure, Resonant scattering and diffraction, Beamline P09, cryogenics, magnetism, crystallography.

Structural transformations of chevkinite group minerals

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The subject of this work is part of a larger project on understanding the processes of mobilization and concentration of Rare Earth Elements (REE) in the Earth's crust. The chevkinite-group minerals (CGM) are dominantly monoclinic REE-Ti-Fe sorosilicates, with REE2O3 contents up to ~50 wt%. Minerals of the group are known from hundreds of terrestrial localities and have also been recorded in lunar and Martian rocks. The crystals of chevkinite-(Ce) studied originated from the pegmatite of Harramosh, Pakistan, and had an average formula: (Ce1.8La0.81Nd0.65Ca0.44)4Fe2+(Fe2+1.02Ti0.77Mn0.14Mg0.10)2.0Ti2.0(Si2O7)2. CGM have complex chemical compositions and complicated atomic arrangements.

The crystal structure of chevkinite-(Ce) is described in the literature in space groups P21/a or C2/m. These variants have different but close symmetry. This varying symmetry appears to be related to temperature, pressure of the formation of the crystals, as well as later alteration processes. Both space groups belong to the 2/m point group, but P21/a is of lower symmetry than C2/m. (1) The factors responsible for a given atomic arrangement have not been discussed in the literature. The aim of this project was to examine these factors and determine transformation pathways. We carried out a series of single-crystal X-ray diffraction measurements on natural and crystals hydrothermally altered in the laboratory.

The crystal symmetry of natural and altered samples was determined as P21/a but the natural crystals were closer to C2/m symmetry than the hydrothermally altered crystals. After single-crystal XRD experiments selected crystals were annealed in an oven or loaded into Diamond Anvil Cell in order to test the influence of high-pressure, high temperature on the structure of chevkinite.

The experiments in the oven were carried out at temperatures ranging from 550 to 1000oC with durations ranging from 6h to 18 days. The changes due to temperature were evaluated by further comparative single-crystal XRD measurements on each of the crystals. Transformations were observed in both directions (from P21/a to C2/m, from C2/m to P21/a) depending on the conditions of annealing.

In the other part of the project, I examined the influence of high-pressure on the crystal structure of chevkinite-(Ce). We carried out a series of single-crystal XRD experiments in DAC up to 5 GPa for selected crystals. High pressure was in all cases promoting the gradual transformation of the crystals from P21/a to C2/m symmetry.

We were able to quantify the changes induced by high-pressure and high temperature statistically by analysing the changes of intensities of reflections forbidden in the C2/m symmetry.

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Classical atomistic simulations of the 10Å phase at high temperatures and pressures

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The 10Å phase, $Mg_3Si_4O_{10}(OH)_2 \cdot nH_2O$, is an important member of the family of dense hydrous magnesian silicates (DHMS) playing a major role in the transport and balance of water in the Earth's upper mantle. A simplified model of the 10Å phase based on the crystal structure of talc has already been successfully investigated by classical atomistic simulations (Wang, 2004). However, more recent experiments show that tetrahedral silicate layers of the real 10Å phase may contain a certain amount of hydrogarnet-type structural defects (Welch, 2006), which would affect its properties, especially at high temperatures and pressures. Here we are using the most recent modification of the ClayFF force field (Cygan, 2021), which can now more accurately account for the bending of Mg-O-H angles in the mineral layers and Si-O-H angles of the hydrogarnet-type defects in classical atomistic simulations. The pressure and temperature dependencies of the 10Å phase properties are calculated in a series of molecular dynamics simulations and compared with a diverse set of available experimental data, including X-ray diffractometry, neutron scattering, IR and Raman spectroscopy.

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Correlation effects on the structure and stability of Nickel Oxides under pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Here, we analyse potential exotic nickel oxides under pressure and the importance of magnetism and localization effects in such compounds. We have performed structure search at 150, 300, and 450GPa using standard DFT, revealing the stabilization of novel compounds and compositions. Given the importance of localization effects on Ni compounds, searches have been complemented with a self-consistent Hubbard U analysis as well as the recently developed rSCAN functional. Our analysis shows that, despite the large pressures involved, self-consistent U is still substantial. It also has a dramatic effect on the stabilization of stoichiometries. Conventional PBE calculations favour novel oxides in paramagnetic states, but LDA+U and rSCAN suggest antiferromagnetic NiO may still be the dominant compound even at pressures as high as 450 GPa. This implies the localisation of d-electrons plays a continued crucial role in nickel oxides. Also, NiO2 is shown to be stable across the pressure range we explored, adopting P ca21 symmetry under 410GPa and P¯3m1 above with all three methods. Ni3O4 is predicted to be stable with PBE and meta-stable with PBE+U and rSCAN functional across the pressure range, undergoes a transition sequence P21c-Cmcm-R¯3m.

The thermal conductivity of liquid Fe-S-Si alloys at 2-5 GPa with implications for the dynamos of small outer Solar System planetary bodies

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The magnetic fields of terrestrial planetary bodies derive largely from the flow of liquid Fe alloys in their cores (e.g., Tikoo & Evans, 2022). Various light elements–S, Si, O, H, C–likely to be present in these cores could affect the processes underlying magnetic dynamo action, such as thermal convection or compositional convection. The physical properties of dominantly Fe cores of terrestrial planetary bodies could be affected by substantial S and Si. For example, the core of asteroid 4-Vesta has an estimated composition of 14.5±1.5 wt% S (Steenstra et al., 2019) and 1-2 wt% Si (Pringle, 2016). Aside from Callisto, all the Galilean moons are thought to contain iron-sulfur cores with pressures near that of this study.

To simulate the conditions of such planetary cores, experiments were performed on Fe-8wt%S-4.5wt%Si in a 1000-ton cubic anvil press reaching 2-5 GPa and 2000 K. Resistivity measurements were made in situ through the electrode mode of Type-S thermocouples, and thermal conductivity was derived using temperature measurements and the Wiedemann-Franz Law (See Figure 1). Similar experiments were performed on Fe-16S-2Si. At 2-5 GPa, an electrical resistivity of 220–270 μΩ·cm and a thermal conductivity of 16-19 W/m/K were found for liquid Fe-8S-4.5Si at ~1700 K.

With these thermal conductivity results, scenarios of the thermodynamic and magnetic history of relevant planetary bodies may be discussed. Using Fourier's Law, the adiabatic heat flux through the cores of these bodies may be estimated. From the magnetic Reynolds number, the maximum thermal convective force through the core of Io such that dynamo action does not occur (as observed) would be on the scale of microwatts per meter squared. Similarly, the effect of S and Si on the age at which the Vestan dynamo shut off may be discussed. The adiabatic heat flux is estimated through the hypothetical Fe-8S-4.5Si core of an exoplanet with a silicate mantle (See Figure 1).

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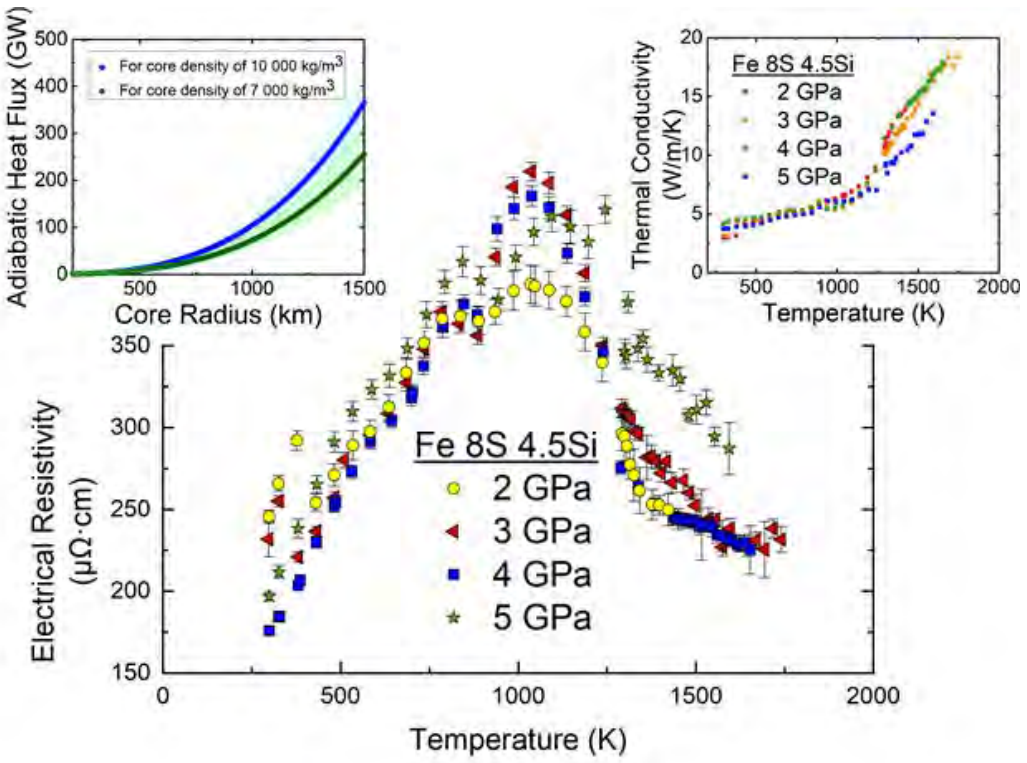
Adiabatic Heat Flux (GW)
500
400
300
200
100
0
500
1000
1500
Core Radius (km)
For core density of 10 000 kg/m3
For core density of 7 000 kg/m3
Thermal Conductivity (W/m/K)
20
15
10
5
0
500
1000
1500
2000
Temperature (K)
Fe 8S 4.5Si
2 GPa
3 GPa
4 GPa
5 GPa
Electrical Resistivity (μΩ·cm)
350
300
250
200
150
0
500
1000
1500
2000
Temperature (K)
Fe 8S 4.5Si
2 GPa
3 GPa
4 GPa
5 GPa

Melting experiments of the system Fe-Si-O under high-pressure with implications for the Earth's core

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Earth's core consists mainly of Fe alloy with some amount of impurities. Silicon and oxygen are likely major impurities in the Earth's core as a consequence of metal-silicate partitioning in the magma ocean early in Earth's history. Robust determination of the crystallising phase relations and thermodynamics of Fe-Si-O liquids are imperative to understand the origin of the geodynamo. After the core was formed, the geodynamo started and generated the early magnetic field, when the wholly liquid core started vigorous convection. The multiple crystallisation events in a liquid core are linked to secular changes in the intensity of magnetic fields. Thus, the generation of strong magnetic fields has been associated with chemical differentiation in the liquid core.

A complete thermodynamic model has yet to be constructed for the system Fe-Si-O in order to fully understand core processes including crystallisation sequence. We conducted melting experiments in the system Fe-Si-O in an internally resistive heated diamond anvil cells (DAC) under high-pressure (P) and temperature (T) conditions. The internally heated DAC stably produces high temperatures (<4000 K) and therefore we are providing precise laboratory measurements. This will resolve inconsistencies observed in laser-heated DAC experiments between different research groups. Melting was judged based on post-mortem textural observation, namely recovered samples are analysed with an FIB-SEM system at the University of Edinburgh.

We will show results of our experiments and discuss the crystallisation sequence of a hypothetical Fe-Si-O liquid core.

Sound velocities in the Lunar Mantle

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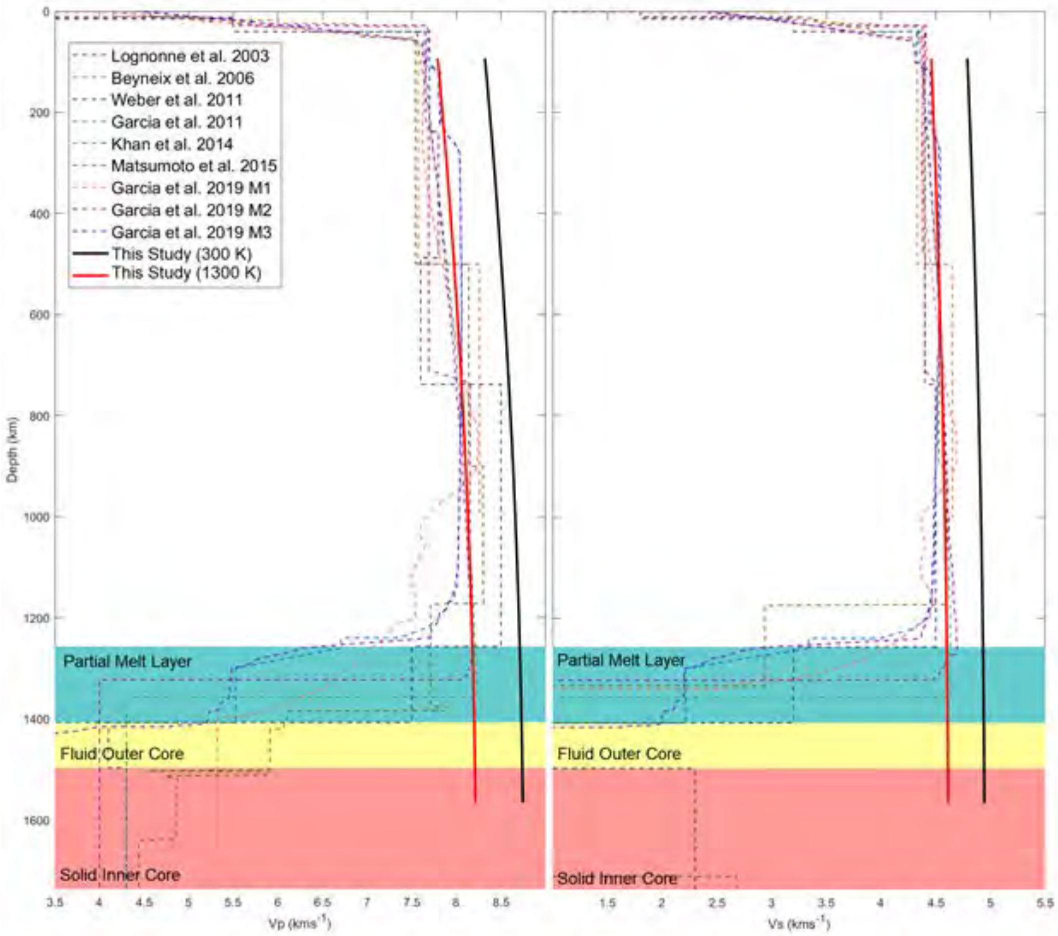
Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The Moon has an interior structure comprised of a crust, a mantle, and a small metallic core (5-6 GPa, 1300-1900 K). Depending on the bulk silicate composition of the Moon, e.g. Taylor Whole Moon (TWM) or Lunar Primitive Upper Mantle (LPUM) (Taylor Planetary Science: A Lunar Perspective 1982; Longhi Geochim Cosmochim Acta 2006), recent experimental and theoretical studies on lunar magma ocean crystallisation have suggested that garnet may play a significant role in the deep lunar mantle, with cumulates containing 0-40% garnet (Jing+ Earth Planet Sci Lett 2022; Kraettli+ ICARUS 2022; Johnson+ Earth Planet Sci Lett 2021; Elardo+ Geochim Cosmochim Acta 2011). While phase relation studies indicate a deep lunar mantle consisting of olivine, pyroxene, and garnet, the compatibility of such an assemblage with seismic models of the lunar interior (e.g. Garcia+ Space Sci Rev 2019; Weber+ Science 2011) is yet untested. Knowledge of the sound velocities of candidate lunar mantle materials are essential in order to interpret lunar seismic velocity models and apply constraints on the mineralogy of the lunar mantle.

Iron-rich peridotitic assemblages were synthesised using the multi-anvil press at pressure and temperature conditions of 6 GPa and 1200 deg C at GRC, Ehime University. The bulk composition of the glass was taken from that of cumulates in the deep lunar mantle found from phase relation studies using a TWM composition (Jing+ Earth Planet Sci Lett 2022). In situ experiments were carried out at BL04B1 at SPring-8 taking simultaneous measurements of ultrasonic interferometry, X-ray diffraction, and X-ray radiography to determine sound velocities at temperature and pressure conditions up to ~ 8 GPa and 1300 K. These sound velocities are interpreted by comparison with lunar seismic velocity models at lunar mantle conditions of 3-4.5 GPa and 1300-1800 K in order to discuss the composition of the lunar mantle.


Lognonne et al. 2003
Beyneix et al. 2006
Weber et al. 2011
Garcia et al. 2011
Khan et al. 2014
Matsumoto et al. 2015
Garcia et al. 2019 M1
Garcia et al. 2019 M2
Garcia et al. 2019 M3
This Study (300 K)
This Study (1300 K)
Partial Melt Layer
Fluid Outer Core
Solid Inner Core
Depth (km)
0
200
400
600
800
1000
1200
1400
1600
3.5
4
4.5
5
5.5
6
6.5
7
7.5
8
8.5
Vp (kms^{-1})
1.5
2
2.5
3
3.5
4
4.5
5
5.5
Vs (kms^{-1})

Hornblende Crystal Populations of Appinites from the Jiagou Mesozoic Intrusion, Southeastern Margin of the North China Craton, and their Genetic Implications

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Igneous rocks are composed of crystal populations, considering that antecrysts are usually present in them. We systematically studied the microtextural feature and chemical composition of hornblende (Hb) grains and classified them into multiple types, which occurred in Jiagou appinites within the Mesozoic intrusion, in southeastern margin of the North China Craton (NCC). Chemical distinctions in Hb grains and major and/or trace element zoning patterns across the Hb crystals characterise four types, i.e., melt phase, eutectic/cotectic, peritectic and xenocryst. Discrepant Hbs belong to different crystal populations genetically, which were formed under different pressure (depth) and temperature (P-T) conditions or magmatic processes, such as fractional crystallisation and magma mixing. Based on further equilibrium thermodynamic calculation, we figured out that the four crystal populations of Hbs are likely to be derived from different magma subsystems or various sources in origin, suggesting that parental magma forming the Jiagou appinites (diorite porphyrite and mafic xenoliths) is the product of magma mixing through complex chambers. The results of genetic mineralogy from different crystal populations of hornblende is of great significance to understand petrogenesis of the Jiagou Mesozoic appinitic intrusions in SE margin of the NCC.

Key words: hornblende; crystal population; appinites; genetic mineralogy; SE margin of the NCC

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Synthesis and Structural Study of Lanthanum Aluminium Hydrides under High-Pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

To obtain high transition superconductors, forming of ternary super-hydrides via chemical precompression at moderate pressure is proposed. Theoretical calculations demonstrated the superconductivity of LaAlH8 with a Tc of 130 K at 100 GPa [1].

We synthesised the lanthanum aluminium alloy by mixing lanthanum and aluminium with an atomic one-to-one ratio at a temperature above 1300 K. The obtained lanthanum aluminium alloy with ammonia borane was laser heated above 2000 K under the pressure of 100 GPa. The results of synchrotron XRD data showed the formation of the LaAl with a space group of Cmcm (a=7.76 Å, b=9.47Å, c=5.77Å). Our initial electric resistance measurements demonstrated the standard metal behaviour down to 14K at 100 GPa. Results of systematic studies on the structure and transport properties of LaAl hydrides under high-pressure will be presented.

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Development of a high-pressure cell for SPS equipment

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The SPS sintering process is a new technology that sinter powders at a uniaxial pressure of about 100 MPa and a temperature of about 2,500°C. Since densification can be achieved at lower sintering temperatures and shorter processing times than conventional sintering processes, it is actively used in the research and development of new functional materials. Furthermore, the combination with high-pressure technology is effective in synthesizing new functional materials, and recently a high-pressure SPS system that combines SPS and high-pressure of about 10 GPa has been developed. By applying ultrahigh-pressure during synthesis, various parameters such as grain growth, structural stability, and chemical reactivity can be adjusted, and densification of nanomaterials, metastable materials, and difficult-to-sinter materials can be expected. However, as in diamond synthesis, synthesis under pressures of 5 GPa or higher is not easy, and the choice must be made between increasing the overall size of the apparatus or reducing the sample volume.

Therefore, the objective of this study is to develop a high-pressure cell that can easily perform SPS sintering at a pressure of 5 GPa without the need for high-pressure specialists.

A supported anvil type with a tip diameter of 4.0 mm was used as the high-pressure generator. This anvil was found to be reproducible for a load of 6 tons up to 873 K. To calibrate the pressure in the sample chamber, the electrical resistance of Bi was measured, and a pressure generation curve was generated for the load pressure.

On the other hand, since plasma is expected to be generated inside the sample during SPS sintering, it is difficult to measure the temperature of the sample chamber directly. Therefore, we sealed a metallic substance that would serve as a temperature indicator inside the sample chamber, installed thermocouples at several radial locations in the high-pressure cell, and created a temperature distribution curve inside the cell from the measured temperature values to estimate the temperature. This high-pressure cell was installed in the SPS apparatus LABOX-300 (Sinterland Corporation), and heating experiments were conducted while varying the pressure in the range up to 3 tons, which is the upper limit.

The sample was a Mg_2Si thermoelectric material that had been synthesised with this apparatus in the past, and the starting material was a mixture of MgH_2 and Si powder.

The sintering characteristics of the sample recovered after SPS sintering were evaluated by microscopic observation and XRD analysis.

Experimental results showed that the samples could be recovered without any problems even when the applied pressure was varied from 500 MPa to 2.4 GPa and heated.

In addition, evaluation of the recovered sample confirmed that the temperature estimated from the temperature distribution curve was almost the same as the temperature inside the sample. Although the load test this time was up to 3 tons due to the limitation of the SPS equipment, it is expected that load tests up to 6 tons can be easily performed.

High-pressure and low-temperature studies of guanidinium iodobismuthate(III)

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Since the first incorporation of metal halide perovskites in solar cells, tremendous progress has been made in their power conversion efficiency. Presently, also perovskite-related materials are intensively studied. The most commonly used divalent cation in these materials is toxic lead, and therefore many efforts have been made to substitute it with more environmental-friendly elements like bismuth or antimony. Here, we present our studies of guanidinium iodobismuthate(III), $[C(NH_2)_3]_3Bi_2I_9$.
At atmospheric pressure and room temperature the compound crystallises in the form of dark red plates of the orthorhombic symmetry Cmcm [1], and the unit cell parameters a = 10.5610(7) Å, b = 14.1272(9) Å, c = 21.1523(10) Å, and V = 3155.9(3) Å^3. The differential scanning calorimetry (DSC) studies, show that the crystal undergoes three successive phase transitions at T_{43} = 177 K, T_{32} = 238 K and T_{21} = 361 K when heating. The single-crystal X-ray diffraction experiments performed at room temperature and at 210 K allowed us to determine the crystal structures of phases II and III. Our results clearly indicate the disorder of guanidinium cations in phase II. The transition to phase III is associated with lowering of the crystal symmetry to the monoclinic space group $P2_1/m$ and with the ordering of all guanidium cations. In phase III the unit cell volume is halved, V = 1546.4(4) Å^3, and the lattice parameters a = 8.6412(9) Å, b = 20.849(3) Å, and c = 8.7110(13) Å.

The high-pressure single-crystal X-ray diffraction experiments and light absorption studies were made in the pressure range to 5.1 GPa. For pressure generation we used a modified Merrill-Bassett diamond anvil cell (DAC) and Daphne Oil 7575 as a hydrostatic liquid. The ruby fluorescence method was used for pressure calibration. Under pressure the crystal undergoes two successive phase transitions at 0.4 and 1.0 GPa. Both transitions are associated with redshift of the absorption edge. The changes in the optical properties of the material are discussed in relation to the pressure-induced modification of the crystal structure.

Acknowledgments: This work was supported by the Polish National Science Centre, Grant Opus 16 No. 2018/31/B/ST3/02188.

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All-Inorganic Perovskites $CsPbBr_3$ and $CsPbCl_3$ under High-Pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Excellent optical and electronic properties of hybrid perovskites make them prospective materials for low-cost, high-performance devices such as light-emitting diodes, photodetectors, and solar cells. However, their practical application is still hampered by long-term stability problems. The solution may be the use of all-inorganic halide perovskites which show better resistance to moisture, temperature and UV illumination, whereas their optoelectronic parameters are comparable to those of the analogous hybrids.[1]

The high-pressure studies of inorganic metal halide perovskites were reported by several groups,[2–3] but all diffraction experiments were performed for powdered samples only, which are not ideally suited for accurate structural determinations for highly pseudo-symmetric materials, nor for spectroscopic studies and visual microscopic observations. Here, we present the results of our systematic high-pressure study of all-inorganic perovskites $CsPbBr_3$ and $CsPbCl_3$. The single-crystal XRD experiments, supported by powder diffraction, microscopic observations, and single-crystal measurements of the optical absorption edge, were performed using a diamond anvil cell (DAC). For dielectric measurements, a beryllium-copper high-pressure cell filled with compressed gaseous helium was used. Special attention was paid to perform all experiments under hydrostatic conditions.

Our study shows that $CsPbBr_3$ and $CsPbCl_3$ undergo various types of pressure-induced transformations: reversible phase transitions, irreversible chemical reactions reducing the dimensionality of the inorganic frameworks, and amorphization. Interestingly, different transformation paths can be realised under moderate pressure, depending on the time scale and method of the pressure applications. We have revealed new phases for both these materials which grow very slowly under the pressure between 0.5 and 1.5 GPa. The complete transformation can take days or even weeks depending on the quality of the sample and external stimuli. The slow-kinetics transformations shed new light on the stress-related response and stability of the all-inorganic perovskite structures. We have also re-examined and clarified the stability regions of the phases observed. The pressure-induced narrowing and widening of bandgaps has been explained by the structural changes combining bond lengths shortening and octahedral tilts. Our study shows the importance of truly hydrostatic conditions for the clarity and correctness of the discussion on the structural and optical properties of these materials. The hydrostatic results can drastically differ from those of the high-pressure experiments performed in nonhydrostatic conditions, systematically affected by the pressure gradients and regions under different anisotropic stress in the sample, which confuse the conclusions assuming a homogeneous stress in a uniform sample.

Acknowledgements: This work was supported by the Polish National Science Centre, Grant Opus 16 No. 2018/31/B/ST3/02188.

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Simulation of Pressure Effects on Small Molecules Confined inside Carbon Nanotubes using Density Functional Theory and Machine Learning Potentials

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Confinement of small molecules within carbon nanotubes has important technological implications and has been the subject of significant research in recent years. Carbon nanotubes are attractive materials for gas storage, transportation and separation due to their high surface area, low weight, and tuneable pore size. Small molecules, such as CO_2, are of particular interest due to their potential use in carbon capture and storage. However, the behaviour of small molecules inside carbon nanotubes is not well understood, and the potential for polymerization of these molecules under confinement remains a topic of active investigation. Our simulations aim to understand the relationship between confinement and pressure exerted on the molecules.

We perform Density Functional Theory (DFT) calculations to obtain accurate molecular energies and forces for the small molecules inside the nanotubes. These DFT calculations serve as the reference data to train our Machine Learning potentials, which enable us to efficiently simulate systems with a larger number of small molecules. We use these Machine Learning potentials to simulate the behaviour of small molecules inside carbon nanotubes under different confinement conditions and pressures.

Our simulation approach provides valuable insights into the confinement and pressure effects on small molecules, such as CO_2, and determines whether polymerization occurs inside the nanotube. Additionally, our study could be extended to other small molecules beyond CO_2, with potential applications in energy storage and gas separation.

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Image created by David Abbasi Pérez @ silicostudio.com

Pressure dependence of dynamics in high-density amorphous ice

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Water is the most normal liquid to us due to its daily presence in our life. However, it has various anomalous properties as a liquid from the scientific point of view. Its properties diverge from a normal liquid's behaviour at supercooled temperatures [1]. Interestingly, according to theoretical studies [2][3] water can exist as two different liquid states. Those liquid states are named as high- and low-density liquids (HDL and LDL). The two-liquid-states model became particularly interesting due to experimental reports on two different amorphous solid states of water: high- and low-density amorphous ices (HDA and LDA) which are suggested to be their corresponding glassy states according to the model. While the glass-liquid transition of LDA to LDL can be studied at ambient pressure (Tg1 = 136 K), both HDA and HDL are stable at elevated pressures. In most of the reported studies, HDA, prepared by compression in a mechanical press, is quench-recovered by adding liquid nitrogen and studied at ambient pressure [4].

In this poster, we provide an overview of recent experiments on HDA's dynamics at ambient and elevated pressures studied by X-ray Photon Correlation Spectroscopy (XPCS) [5]. XPCS is a powerful technique to determine viscous dynamics in glasses using coherent X-rays. Thereby, scattering intensity fluctuations obtained from sample heterogeneity are correlated over time. Measuring in small-angle geometry, we determine a collective motion and the related relaxation time of the system. Our high-pressure cryogenic setup allows us to investigate the glass transition in high-density amorphous ice (HDA) at elevated pressure.

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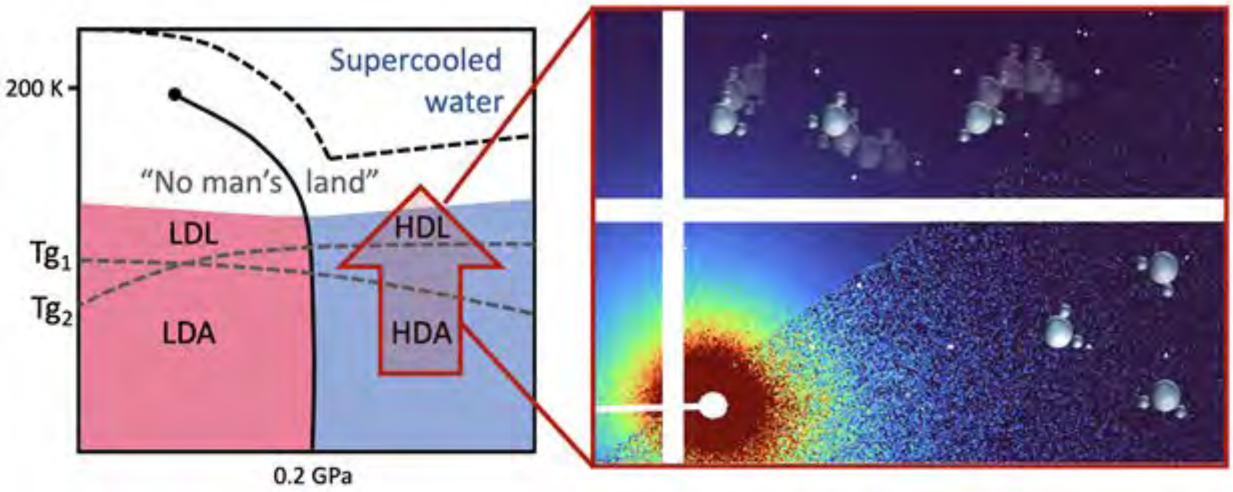

Characterising C-H demixing and Hydrogen metallization in Warm Dense Matter conditions

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Giant and ice-giant planets have dominated the numbers in the ever-increasing list of exoplanets. The conditions in the interiors of these planets are at the low-temperature end of the warm dense matter (WDM) regime. The study of pressure-induced liquid–liquid phase transition from molecular hydrogen to a metallic phase is very important for understanding the interiors of these planets, as well as the physical and chemical behaviour of materials at high-pressures and high temperatures [1]. The exceptionally strong magnetic fields of gas giant planets can be explained by the condensed metallic hydrogen in the interior of these planets [2]. Metallic hydrogen near boundary regions between their gas envelopes and ice cores is expected to be responsible for the dominant contributions to the unusual fields of the ice giants [3].

It is predicted that in the high-pressure and high-temperature environment of these ice-giant planets, methane present in their interiors will form polymeric hydrocarbon chains [4]. Subsequently deeper towards the core, it will dissociate into carbon in form of diamond and metallic hydrogen. The formation of diamond under these WDM conditions has already been demonstrated in experimental conditions [5]. Meanwhile, conclusive observation of metallic hydrogen in these conditions has been quite challenging.

We will present an experimental approach where we propose to observe and characterise hydrogen metallization in dynamically compressed hydrocarbons, by using the spectrally resolved X-ray scattering method in carbon-hydrogen phase separation regime. With the help of time-dependent density functional theory (TDDFT) calculations [6], and scattering spectra collected at the European X-ray Free-Electron Laser, we give an outlook of future experiments, and canvass the possibility of investigating the presence of liquid metallic hydrogen using the rep-rated drive laser systems in future experiments at EuXFEL.

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Spacer Dependent and Pressure Tuned Structures and Optoelectronic Properties of 2D Hybrid Halide Perovskite

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Compared to the 3D counterparts, 2D hybrid organic-inorganic halide perovskites (HOIP) exhibit enhanced chemical stabilities and superior optoelectronic properties, which can be further tuned by the application of external pressure. Here we report the first high-pressure study on CMA2PbI4 (CMA = cylcohexanemethylammonium), a 2D HOIP with a soft organic spacer cation containing a flexible cyclohexyl ring, using UV-visible absorption, photoluminescence (PL) and vibrational spectroscopy, synchrotron X-ray microdiffraction, aided with density functional theory (DFT) calculations.

Substantial anisotropic compression behaviour is observed as characterised by unprecedented negative linear compressibility along the b-axis. Moreover, the pressure dependence of optoelectronic properties is found to be in strong contrast to those 2D HOIP with rigid spacer cations. DFT calculations help to understand the compression mechanisms leading to pressure-induced bandgap narrowing. These findings highlight the important role of soft spacer cations in the pressure-tuned optoelectronic properties and provide guidance to the design of new 2D HOIPs.

Pressure-Induced Emission Enhancement of π-conjugated Charge-Transfer Materials with Different Molecular Stacking

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Among the large variety of organic luminophores, organic charge-transfer (CT) molecules stand out from peers for their versatile applications in biological imaging, specific matters detection, and the next generation of organic light-emitting diodes. However, CT materials suffer from intrinsically limited emission efficiency caused by insufficient spatial overlap between excited state electrons and hole's wave functions, which hinders their practical application. High pressure has been regarded as a valid technique to promote organic fluorescence, yet the enhancement of CT emission by pressure has not been realised. Here, we report the successful pressure-induced emission enhancement in two typical CT molecular materials: 6-lauroyl-N,N-dimethyl-2-naphthylamine (Laurdan) and 6-propionyl-N,N-dimethyl-2-naphthylamine (Prodan). Due to the difference on the lengths of the alkane chains, the two molecules adapt different packing arrangements in crystals. In the Laurdan crystal, the molecules adapt a slipped co-planar parallel π-stacking mode. Under compression, the Laurdan crystal exhibits distinct emission enhancement up to 4.1 GPa accompanied by a shift in the emission colour from blue to cyan. Further experimental and theoretical characterizations demonstrate that the π-π interplanar distance between Laurdan molecules is reduced during pressurization, and intermolecular wave function diffusion is improved simultaneously, which results in local excitation promotion in the multi-molecular excited states, thus the enhanced emission. Thereby we put forward a new multimer mechanism of emission enhancement in molecular aggregates. Comparatively, the Prodan molecules are stacked in a typical herringbone π-stacking mode in crystal, therefore the luminescent response to pressure varies distinctly as well. The emission of Prodan crystal is enhanced up to 1.1 GPa, and the magnitude of enhancement notably increases up to 3 times. Our findings not only provide new insights into CT emission modulation but also pave the way for the further design of high-performance aggregated CT materials.

Extreme pressure-sensitivity of electrical resistivity in Pt(bqd)²

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Organic materials show a tremendous diversity of electrical conductivity behaviour and have been found to exist as semiconductors,[1] metals[2] and superconductors.[3] Because of their layered crystal structure, these materials exhibit anisotropic properties. In combination with relatively weak Van Der Waals interactions between the molecules these result in highly anisotropic compressibility of the materials.

In this work we present recent hydrostatic and uniaxial studies of a transition metal complex with an organic ligand - the bis(1,2-benzoquinonedioximato)platinum(II) (or Pt(bqd)²). One of the key discoveries is that when this material is subjected to a moderate uniaxial pressure of approximately 150 MPa it demonstrates nearly 5 orders of magnitude decrease in electrical resistivity. The cycling of pressure confirms reversibility and repeatability of the ρ vs P data.

Such extreme sensitivity of electrical resistivity to external pressure can be explained by the π-stacking arrangement of molecules when material is grown as a thin-film with the preferred needle orientation. Compared to continuous-lattice materials, intramolecular interactions in Pt(bqd)² are weak and do not lead to high intermolecular repulsion when molecules are pressed closer. This results in high-pressure dependence on electronic properties, thus making this class of materials potential candidate for novel data storage applications.[2,3,4]

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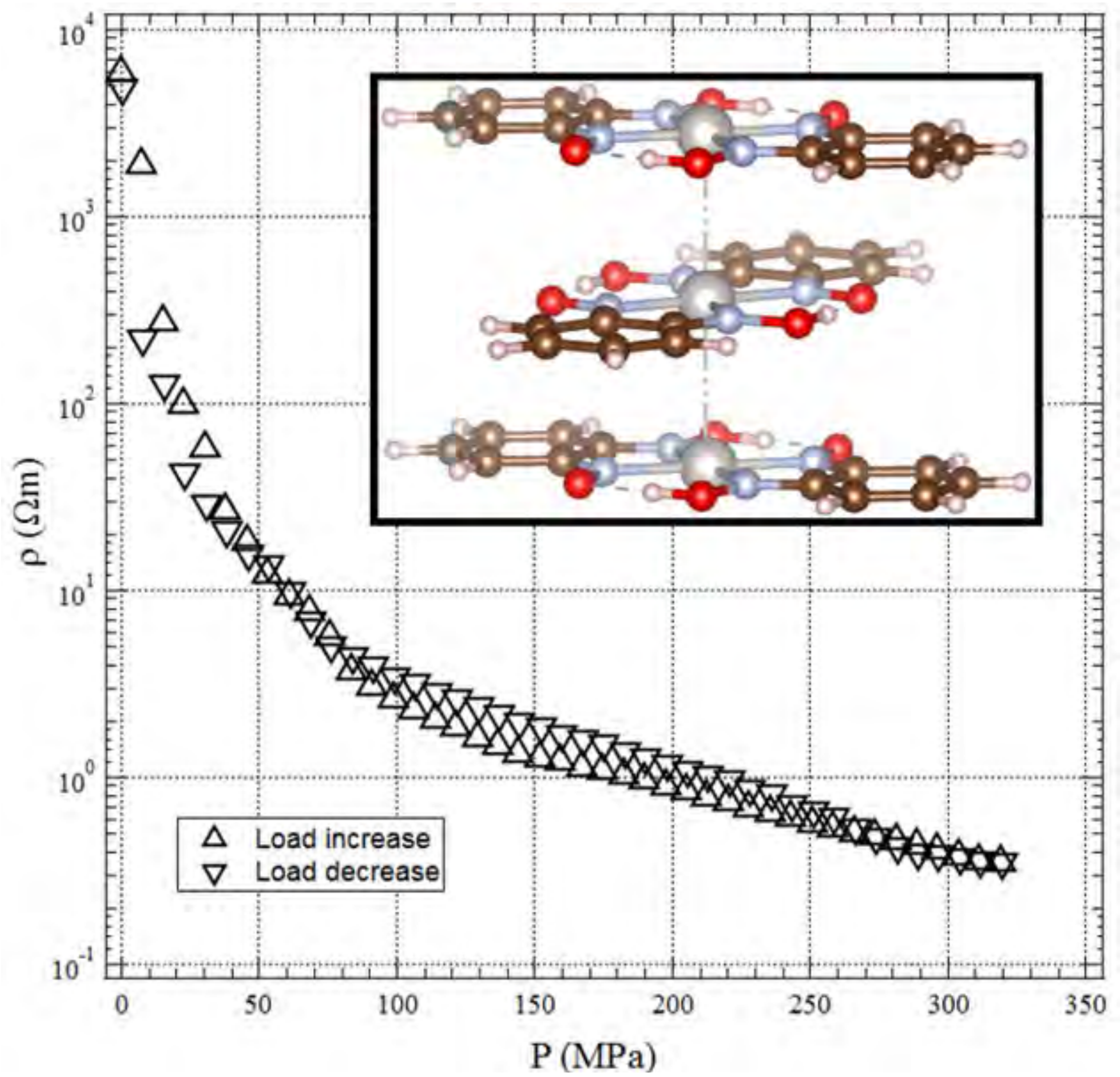

Fig.1 Resistivity vs pressure in thin-film Pt(bqd)². The insert shows crystal structure with Pt-Pt one-dimensional molecular stacking.

Use of novel composite materials in construction of non-metallic high-pressure cells

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Non-metallic high-pressure cells are required in a number of physical property measurements from magnetic ac susceptibility to studies in high pulsed magnetic fields, where the heating or signal screening effect from Eddie currents are an obstacle. We investigate the applicability of several state-of-the-art fibre-reinforced polymers for design and fabrication of high-pressure cells using finite element analysis and experimental testing. Performance and failure modes for the key components of the cell working in tension and in compression were evaluated and the material properties critical for high-pressure applications were established. Finite element analysis was used for optimising designs of high-pressure cells made of composite materials.

A miniature non-metallic diamond anvil cell for magnetic ac susceptibility measurements in Quantum Design instruments was designed and built. The cell is short and symmetric and fits into the 9 mm bore of the QD MPMS, and can also be used with the VSM option of the QD PPMS instrument. Forces in excess of 3 kN and pressures over 5 GPa were generated using different materials for the construction of the cell. AC susceptibility data collected on Dy2O3 demonstrate the performance of the cell in magnetic property measurements and confirm that there is no screening of the sample by the environment which typically accompanies the use of conventional metallic high-pressure cells in oscillating magnetic fields.

Stoichiometric Determination of Clathrate-like Yttrium Hydride under Megabar Conditions

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The pathway to high-temperature superconductivity, approaching room temperature, has been demonstrated by multiple hydrogen-dominant alloys under high-pressure conditions. Despite this, direct measurements of the hydrogen sublattice in a diamond anvil cell have been elusive, even at near-ambient pressures. Binary rare-earth metal-hydride systems were predicted to be excellent candidates for achieving room temperature superconductivity, with early predictions of both LaH_{10} and YH_{10} both achieving higher superconducting critical transitions than 0°C, with well-understood, conventional, superconducting mechanisms. Experimental measurements confirmed high transition temperatures, but below theoretical predictions with maximum superconducting transition temperatures of 260 K at 188 GPa and 262 K at 182 GPa for LaH_{10} and YH_9 respectively. In this work, we perform X-ray diffraction (XRD), X-ray absorption (XAS), and X-ray emission spectroscopy (XES) up to 200 GPa and measure coordination in the clathrate-like yttrium hydride system synthesised in situ in hot hydrogen-rich pressure-transmitting medium. We find that at megabar pressures XES remains a viable candidate to discern stoichiometries through observation of shifts of valence-to-core (VTC) transitions. As a result of changes to the PDOS accompanying increased coordination, YH_2, YH_3, and YH_4 can be distinguished from experimental XES measurements in this work. Furthermore, tetragonal distortions of the Fm-3m YH_3 to an I4/mmm phase were observed through XRD and X-ray spectroscopic techniques at 194 GPa.

Phase transitions and electronic properties of Fe2O3 under laser compression by ultrafast in-situ X-ray absorption spectroscopy

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Understanding the structural changes of hematite (α-Fe2O3) under extreme conditions of pressure and temperature is crucial for gaining insights into the physical properties of planetary interiors such as Earth and super-Earths (2 to 10 times more massive).

At ambient conditions, hematite is a rhombohedral structured antiferromagnetic insulator [1,2]. Its high-pressure behaviour has been largely studied over the past decades using static compression [1,3,7]. Several phase transitions were reported but particular attention was given to the structural, electronic and magnetic changes occurring ∼50 GPa: a 10% volume cell drop accompanied by a change of crystal symmetry corresponding to a distorted perovskite (ζ-Fe2O3) [3], a Mott insulator-metal transition and the collapse of the iron magnetic moments corresponding to the high-spin (HS) to low-spin (LS) electronic phase transition [3-7]. However, the exact nature of the phase transition in this pressure area remains controversial [4,6,7]. The question of which transition drives the other one, electronic or structural, is still under debate.

Here, we report ultrafast time-resolved X-ray Absorption Near Edge Spectroscopy (XANES) measurements obtained on laser-compressed Fe2O3 at the High-Power Laser Facility (HPLF) of ESRF-ID24 beamline [8]. Our XANES data provide information on time-resolved structural transformations by showing changes in the pre-edge, the white line and the 1st Extended X-Ray Absorption Fine Structure (EXAFS) oscillation within hundreds of ps after the shock breakout from the samples. More severe spectral changes are observed at longer delays between the X-ray probe and the shock, during its thermodynamic release. We will present a detailed time-resolved study of the XANES changes as a function of pressure and temperature, along the Fe2O3 Hugoniot thermodynamic path and release. For further understanding of the XANES features, preliminary FDMNES [9] and Quantum Espresso [10] ab-initio calculations will also be presented.

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Pressure-induced phase transitions and Equations of State for a luminescent arylacetylide-gold(I) compound.

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Formation of polymorphs or solvates of new two-coordinated gold(I) complexes has drawn considerable interest in recent years [Mirzadeh2020]. Numerous studies in this area revealed that aurophilic interactions of gold atoms are sensitive to environmental factors, such as counterions, static external pressure or solvent molecules which may significantly change their luminescent properties both in solution and in the solid state, broadening the range of their potential application. We decided to attempt using increased pressure as means to tune the structural and luminescencent properties of a weakly luminescent 1-pyrene-acetylide-gold(I)-triethylphosphine[Głodek 2018], further denoted as PirEt. The compound undergoes ligand scrambling in solution, which results in formation of stable piezochromic and barochromic crystals that contain [ArCOCC]2Au- (Et3P)2Au+ homoleptic species.

High pressure, single crystal X-ray diffraction experiments of these crystals were carried out both in-house and at the ESRF ID15B synchrotron beamline with external pressure varied between 1 atm and 9 GPa. Face transition from triclinic to monoclinic form was observed above 0.6 GPa. Experimental Equations of States were acquired for both monoclinic and triclinic form and compared to theoretical Equation of States obtained from periodic DFT calculations. The discrepancies between the experiment and theory will be discussed with respect to the computational method. In both crystal forms, the infinite Au...Au...Au chains were observed and conserved. Solid-state luminescence spectra at increased pressures were also recorded and correlated with atomic rearrangements and the calculated band structure changes.

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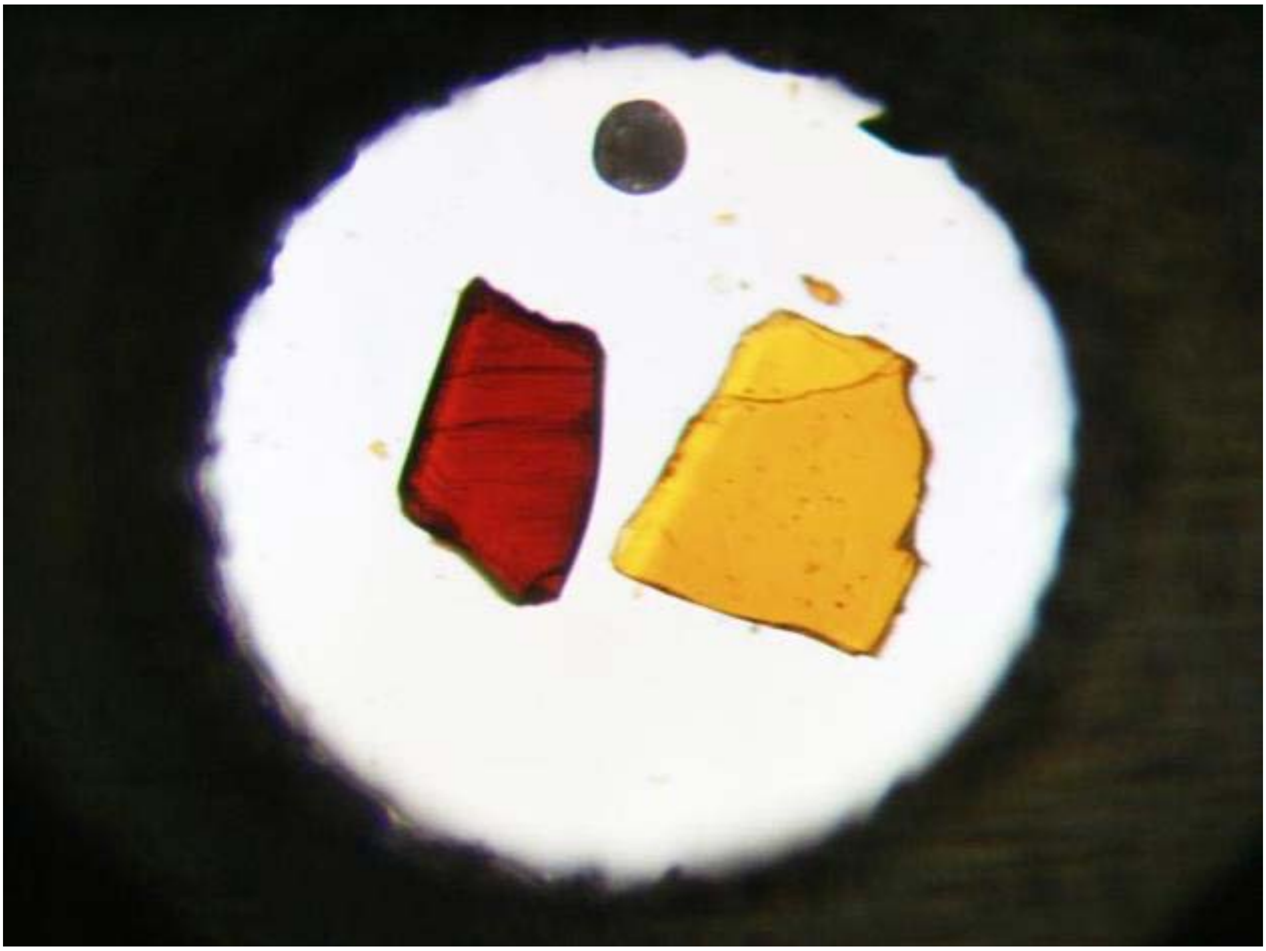

Phase transition and sound velocity study of shock loaded CaF2

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Fluorite, CaF2, is a model ionic solid and prototypical structure type in condensed matter physics, undergoing pressure-induced phase transitions to highly coordinated AX2 structure. In this study, the shock-induced phase transitions in CaF2 were investigated by plate impact experiments carried out with the gas guns at Soreq NRC and NRC Negev and powder gun at Soreq NRC. Single-crystal samples in (111) and (100) orientations were shock loaded to pressures between 7 GPa to 45 GPa. The particle velocities were probed by VISAR and PDV. The wave structure evolution measured in samples of different thicknesses allowed us to extract the shock velocity and sound velocity in the shock compressed material. We observe evidence of a transition to a high-pressure phase, where the phase transition seems to be sensitive to pressure and strain-rate.

Thermal conductivity of deep earth minerals using high pressure-temperature time-resolved powder X-ray diffraction at European XFEL.

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Large scale dynamics within the Earth are the result of cooling. Heat is transported towards the surface by large scale convection in the mantle and in the core, and by conduction across the thermal boundary layers at the core-mantle boundary (CMB) and the lithosphere. There is a range of estimates for the thermal transport properties, e.g. thermal conductivity (k) in the lower mantle ranges between 4 and 16 W/m K, resulting from a lack of consensus on how to represent the pressure (and temperature) dependence of k; different models yield very different extrapolations.

A three-pronged approach is here established to study thermal conductivity of deep earth minerals at CMB conditions.

(i) Generating high-pressure and high-temperature states of matter in a diamond anvil cell (DAC) and resolving crystallographic changes in the sample via powder XRD. HED at European XFEL is the only facility at present that has sufficiently high X-ray energy coupled with MHz pulse trains to perform time resolved measurements of heat flow in high-pressure samples heated by XFEL pulse trains. The femtosecond FEL pulses generate a unique thermal disturbance in bulk matter at a definitive time point, providing an idealised starting point for thermal relaxation.

(ii) Powder XRD analysis can be carried out by utilising the different timescales of XRD and X-ray absorption. The AGIPD detector at HED beamline determines the relaxation dynamics and heat flow. The first X-ray pulse is used to collect a diffraction image of the unexcited state of the sample. The next X-ray pulse probes the heated state of the sample 222 ns after first excitation (at 4.5 MHz), before heating the sample again and the step is sequentially repeated allowing the study of cumulative heating events.

(iii) Finite element modelling studies allow the determination of thermal parameters such as thermal conductivity and expansivity. Utilising volume change with temperature in a sample, which can be extracted from the diffraction data, a primary model can be made. Temperature dependent thermal conductivity is fitted to the data. Beam energetic data is integrated into the Finite element modelling to dynamically model the fluctuations in the intensity of energy pulses.

Investigating the iron phase diagram utilizing MHz diffraction at the European X-ray free electron laser

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The opening of the European X-ray Free Electron Laser near Hamburg (EuXFEL) offers promising new experimental opportunities for studying materials under high-pressure conditions. It can deliver X-rays with energy up to 25 keV and the brightness is so large, that a single X-ray pulse exhibits enough intensity to generate a diffraction image and enough energy to heat materials. In combination with the 4.5 MHz (220 ns separation) rate of pulse delivery, and new detector technologies (AGIPD), the EuXFEL enables unique capabilities for time resolved diffraction experiments.

Coupling these new capabilities with the diamond anvil cells, enables the study of materials under planetary conditions with unprecedented time resolution, possibly overcoming previous issues with chemical reactions between the sample and its environment as well as the study of transient phenomena such as phase transformations.

We will present the first results and challenges of experiments investigating the iron phase diagram under extreme conditions at the “High Energy Density” (HED) instrument of the European XFEL. We will focus on detectability of the phase transformations in iron (bcc, fcc, hcp and melt) as well on the metrological challenges with the experiment due to the short experimental runs.

Innovative Design for Multimegabar Diamond Anvil Cell†

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Most diamond anvil cells (DAC) are prone to shear instability due to imperfect fits between piston and cylinder, or in kinematic mounts of plate cells. To overcome this limitation, a novel design has been developed which incorporates a rigid, shallow "pan" and a press-fit "lid" that is elastically deflected in the centre. This design has been successful in achieving multimegabar pressures without any occurrence of lateral shear in the gasket.

In the earlier version of this design, three central screws and gears were used to deflect the lid. However, the latest design employs an additional plate that operates on a "seesaw" principle without gears for central compression, resulting in a simplified design.

Additionally, this new design provides easy lateral and tilt adjustment of the anvils, and pressure can be changed with a single screw while observing the sample and gasket under the microscope.

This technique also eliminates the need for high precision machining, which is essential in conventional piston-cylinder types. In this poster, we will present the design of this novel DAC in details, as well as some preliminary results obtained at pressures in the multimegabar range.

†Paper to be published by R. Boehler

Triple Coil Setup for Studies of Magnetic Properties at High-Pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

In the past years, Almax easyLab developed the Diacell® ChicagoDAC in collaboration with Prof. Daniel Silevitch's group [1]. This diamond anvil cell (DAC) is dedicated to high-pressure – low temperature electrical resistivity measurements.

Here we present a new application of the Diacell® ChicagoDAC. We have developed a coil setup to carry out magnetic measurements at low temperatures and high-pressures. This new development makes this DAC a very powerful tool for studying both the electrical and magnetic properties of materials at high-pressures. The Diacell® ChicagoDAC has the added advantage that the pressure can be tuned at low temperatures using a gas membrane – which would allow establishing direct correlations between electrical and magnetic phases without warming up to room temperature.

In this poster, we will present the experimental details of the setup as well as our results obtained on the half-metallic Heusler alloy – Fe2MnSi in the pressure range 0–9 GPa and temperatures 3–270 K. The Heusler alloy has a ferromagnetic transition on the Fe site at TC = 220 K with an antiferromagnetic component at TN = 67 K on the Mn site [2,3], making it an ideal test candidate for this new application.

The simplicity of the new setup and the promising results indicate that the approach, with some optimization, will be developed towards a rapid, robust and highly sensitive technique for accessing magnetic transitions at high-pressures in a wide range of materials. The approach may also potentially extend ambient pressure magnetization studies to systems where magnetic signals are extremely low, such as thin films and 2D materials, as well as topological heterostructures.

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High-Pressure Investigation of the I-N System

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The pursuit of novel nitrogen-based compounds is of high importance with regards to technological materials–including as high energy density materials, superhard materials–as well as for building empirical knowledge on chemical rules in simple binary systems. Nitrogen's behaviour has been investigated at high-pressures with all but one class of elements in the periodic table: halides. This might appear as surprising given that these unique elements are expected to give radically different results than those observed with previous binary compounds. Indeed, halides have among the highest electronegativities, enabling unique chemical behaviour with nitrogen.

In search of iodo-nitride compounds, a laser-heated diamond anvil cell containing a mixture of iodine and nitrogen precursor materials was taken to 70 GPa and studied with single-crystal X-ray diffraction. Up to this pressure value, no iodine-nitrogen solids were found, in agreement with density functional theory calculations [1]. However, what was found was a new high-temperature, high-pressure form of pure iodine for which its structure was solved and refined (Figure 1).

After determining its structure, the pure iodine allotrope was decompressed to establish its equation of state and stability domain. The new compound was found to be recoverable down to 25 GPa after which only the known orthorhombic Cmce phase is present [2].

In the coming weeks, further investigation into the I-N system will be conducted up to 130 GPa, and exotic compounds, such as an IN6 solid with hypervalent iodine atoms, expected to be formed.

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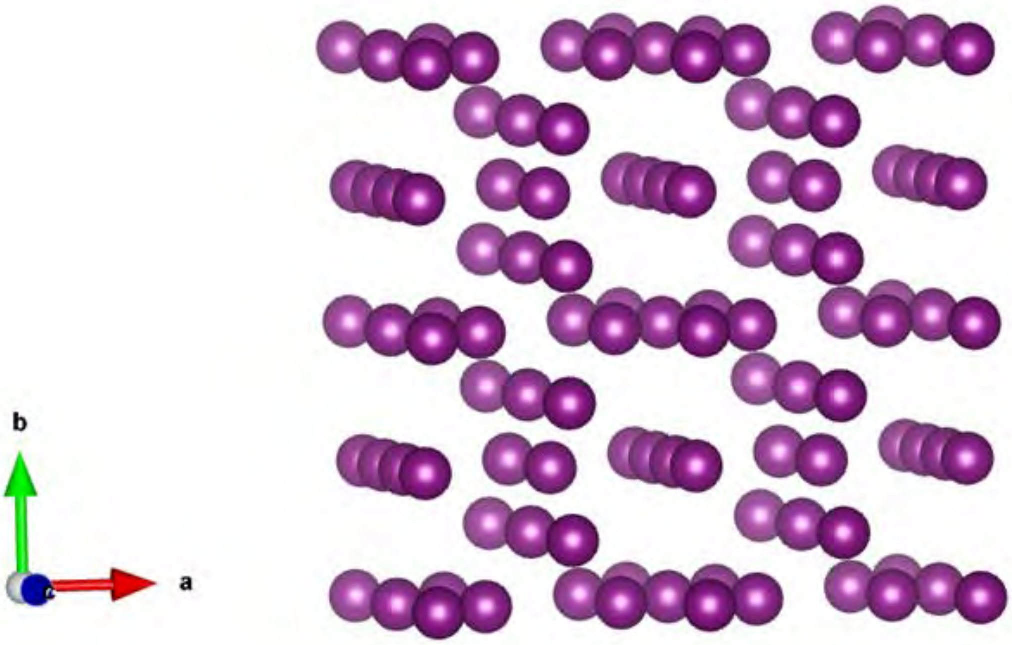

Electrical conductivity of clinochlore at high- pressure and temperature

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Chlorite containing 13 wt.% water in its structure, is one of the most important hydrous minerals in the subducting slab and mantle wedge. The electrical conductivity of clinochlore was measured at pressures of 1.0–4.0 GPa and temperatures of up to 1273 K using impedance spectra. Electrical conductivity of clinochlore samples showed complex features before dehydration, during dehydration and after dehydration. The electrical conductivity results indicate that several different stages corresponding to different conduction mechanisms. A new dehydration model for clinochlore under high-pressure and temperature were proposed. The pores inside clinochlore and the recovered sample after the conductivity measurements were analysed using X-ray computed tomography. Combined with the geothermal gradient, the high-conductivity anomalies observed at depths of 150–200 km in geophysical investigations were interpreted using our experimental data.

Equation of state and diamond formation kinetics of C-H-O mixtures under ice giant interior conditions

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

According to current models, planetary ices (water, ammonia and methane) make up the majority of the interiors of ice giants like Uranus and Neptune. Those interiors are defined by Mbar pressures and are thought to give rise to peculiar phenomena, like the formation of diamonds from de-mixed carbon in the planets' deep layers or superionic water [1, 2]. Still, considerable uncertainties remain when modelling the giants' internal structures to match the observed properties (mass, radius, gravitational field etc.) [3, 4]. Improving these models demands a better knowledge of the equation of state (EOS) of mixtures composed of planetary ice elements at relevant pressures, e.g., to predict the mixing of ices inside the giants [3]. Using laser-driven shock compression, this work presents EOS data for C-H-O compounds with varying carbon to water ratios, namely cellulose acetate ($C:H_2O$ ratio of 1.25) and polylactic acid ($C:H_2O$ ratio of 1.5), to see how a change in carbon concentration influences the equation of state as well as nanodiamond formation, the latter being a result of exposing the compounds to ice giant-like conditions.

The results add to the work done on polyethylene terephthalate ($C:H_2O$ ratio of 2.5) which demonstrated that oxygen promotes nanodiamond formation [5, 6, 7]. That is, diamond could be detected in C-H-O targets in a pressure-temperature region on their principal Hugoniot where no diamond formation was observed for polystyrene $(C_8H_8)n$ [1, 5]. The data also suggest that a higher initial carbon concentration in a mixture leads to a greater nanodiamond yield. These insights are a step towards more accurate interior models and provide constraints for future experiments on C-H-N-O compounds under ice giant conditions as well as a future space mission to Uranus envisaged by NASA [8]. Since ice giants make up the majority of observed exoplanets, a better understanding of our Solar System's giants can bring fundamental insight into how this significant class of planets form and evolve [9]. Finally, it supports the efforts for relevant applications on Earth, e.g. lab grown nanodiamonds which have a plethora of potential use cases [10].

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MHz X-Ray Diffraction and X-Ray-Heating in the Diamond Anvil Cell: a Metrology Study on Fe

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The European X-ray Free Electron Laser (EuXFEL) is a new type of facility characterised by a high brilliance, a MHz pulsed X-ray beam with short pulses below 50 fs. The high Energy beam can be used to locally heat samples and acquire diffraction patterns simultaneously, allowing for a new type of pump and probe experiments at X-ray sources. The High frequency rate (up to 4.5 MHz) enables a high-speed study of temperature-induced phase transitions in diamond anvil cells.

The use of such capabilities for high-pressure experiments, however, are in their infancy. For instance, the metrology of temperature increase in X-ray pulsed heating experiments remains to be understood. Indeed, the diffraction pattern produced by each pulse probes the state of the sample as it is cooling down from the previous pulse, and this should be quantified. In addition, it is unclear whether kinetics can affect the measured material properties at such short time scales.

Here, we present a study of pure Iron compressed in diamond anvil cells at the EuXFEL. The low-pressure phase diagram of Iron is well constrained, enabling a comparison of our results with literature data. All three bcc, fcc and hcp structures are observed depending on pressure and temperature and we confirm that, in fact, transitions to the fcc phase and melting can be induced by X-ray heating at the timescale of the experiment.

Known equations of state of the various Iron phases can be used to constrain pressure and temperature in the samples. However, in many cases, the temperatures measured on the diffraction pattern for phase transitions are lower than that of literature, which can be explained by the delay between the pump and probe X-ray pulses.

This presentation will focus on those results, the effect of thermal pressure, and how finite element models reproducing the experiments can help understanding the metrology of X-ray heating experiments at the EuXFEL.

This work is the result of experiments performed under the EuXFEL 3063 community proposal.

The effect of pressure on superconductivity in AuxPd1-xTe2

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Type-II Dirac semimetal PdTe2 is a type-I superconductor with a transition temperature of ~1.6 K at ambient pressure. While the bulk superconducting property is conventional, the superconductivity of the surface sheath observed in an ac-susceptibility measurement at magnetic fields is unusual and has yet to be explained [1]. At high-pressures, the Tc has a maximum of ~1.9 K around 1 GPa, and the anomalous behaviour of surface superconductivity remains up to ~2.5 GPa at least [2]. Furthermore, the effects of Au- [3], Cu- [4], and Pt- [5] doping have been shown in several studies: the enhancement of superconductivity [3, 4] and the transition to a Type-II superconductor induced by the slight doping of Pt [5]. However, research on the effects of pressure on the superconductivity of doped materials has not been widely conducted. Recently, we have performed an experimental investigation for polycrystalline samples of Au-doped PdTe2 (AuxPd1-xTe2) under high-pressure. In Au0.35Pd0.65Te2, the transition temperature is 4.6 K at ambient pressure consistent with the literature value [3] and increases slightly up to 4.7 K at around 0.6-0.8 GPa, although its pressure variation is small. Results of dc magnetization and heat capacity at magnetic fields show that Au0.35Pd0.65Te2 is a Type-II superconductor. In this contribution, we will present the superconducting properties of the Au-doped material and discuss comparing it with PdTe2 and other doping materials.

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Pressure Effects on the Specific Heat of the Thermoelectric Compound InTe

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Indium telluride (InTe) has received much attention due to the potential application for the thermoelectric material [1]. InTe crystallises in the tetragonal TlSe-type structure at ambient conditions with mixed valent formula In^1+In^3+Te^2−_2 [2]. The trivalent In^3+ ions are tetrahedrally coordinated with four Te^2− ions forming edge-sharing tetrahedra chains, whereas the monovalent In1+ ions reside in tunnels formed by the chains and are surrounded by eight Te^2− ions [2]. The In^1+ ions are vibrating with an anharmonic large amplitude (rattles), leading to low lattice thermal conductivity through a strong anharmonic phonon-phonon interaction [1]. The rattling motion of In^1+ ions manifests itself in the broad double peaks at 3.5 K and 8 K of the specific heat C divided by T^3 [3]. The double peak structure of C/T^3 is not consistent with one peak structure calculated from the phonon spectrum for a pressurised unit cell under P = 3 GPa [3]. At 3.6 GPa, an isostructural electronic phase transition has been observed by Raman and X-ray diffraction measurements [4]. At 6 GPa, the tetragonal phase changes to a face-centred-cubic (fcc) phase [5]. The structural transition is proceeded by a transition at about 5 GPa from a semiconducting to metallic state due to the valence modification from In^1+ and In^3+ to In^2+ [6, 7]. The fcc phase exhibits superconductivity below 3.5 K [7].

To unveil the relation between the rattling motion of In1+ ions and electronic phase transition in InTe, we have measured the specific heat and electrical resistivity from 0.5 to 20 K under pressures up to 7.1 GPa. We used an AC calorimetry which enables us to measure the absolute value of C even under high P [8]. Figure 1 shows the temperature dependence of C/T^3 under various P. For P = 0, a broad shoulder at 3.5 K and maximum at 8 K of C/T^3 reproduce the reported data [3]. The shoulder disappears by applying pressure up to 1 GPa. With further increasing P up to 3.6 GPa, the maximum is suppressed and shifts to high temperature up to 14 K. This feature means the increase of the rattling energy as predicted by the theory [3]. For P ≥ 3.6 GPa, a pronounced step-like anomaly in C/T below 2 K has been observed as shown in the inset and is attributed to the superconducting transition. Therefore, this result reveals that the bulk superconductivity emerges concurrently with the electronic phase transition.

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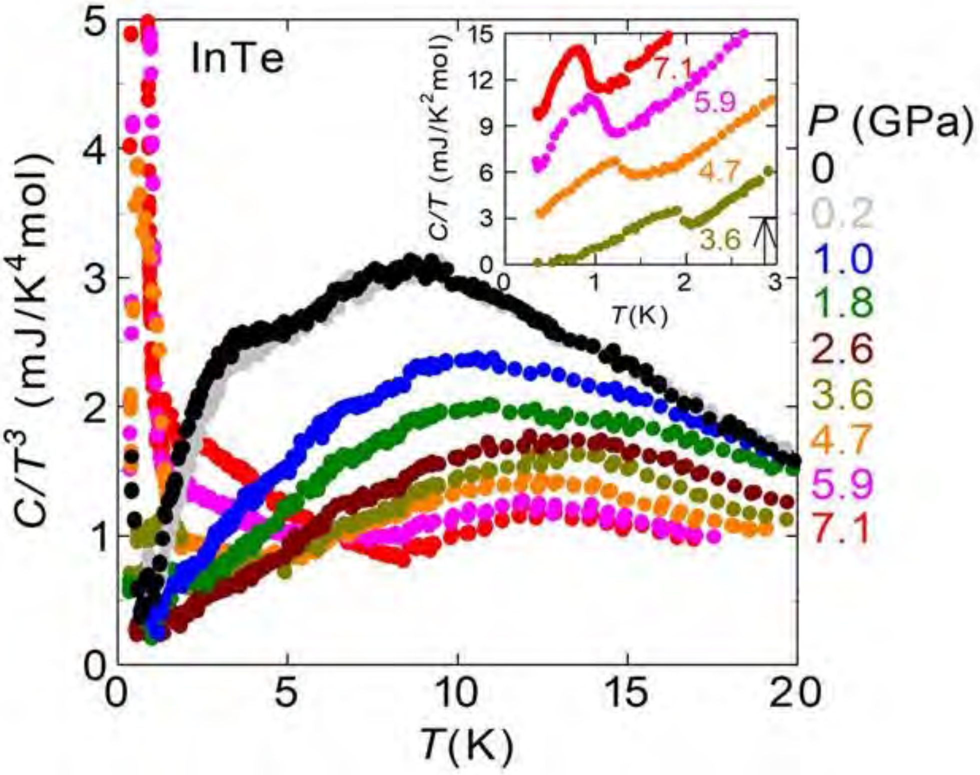

Fig. 1 Temperature dependence of C/T^3 of InTe under various pressures up to 7.1 GPa. The inset shows the temperature dependence of C/T for $P \geq 3.6$ GPa. The data sets are shifted upward consecutively by 3 mJ/K^{2}mol for clarity.

Superhard boron carbide: new insights into anomalous dynamic failure and how to reinforce it

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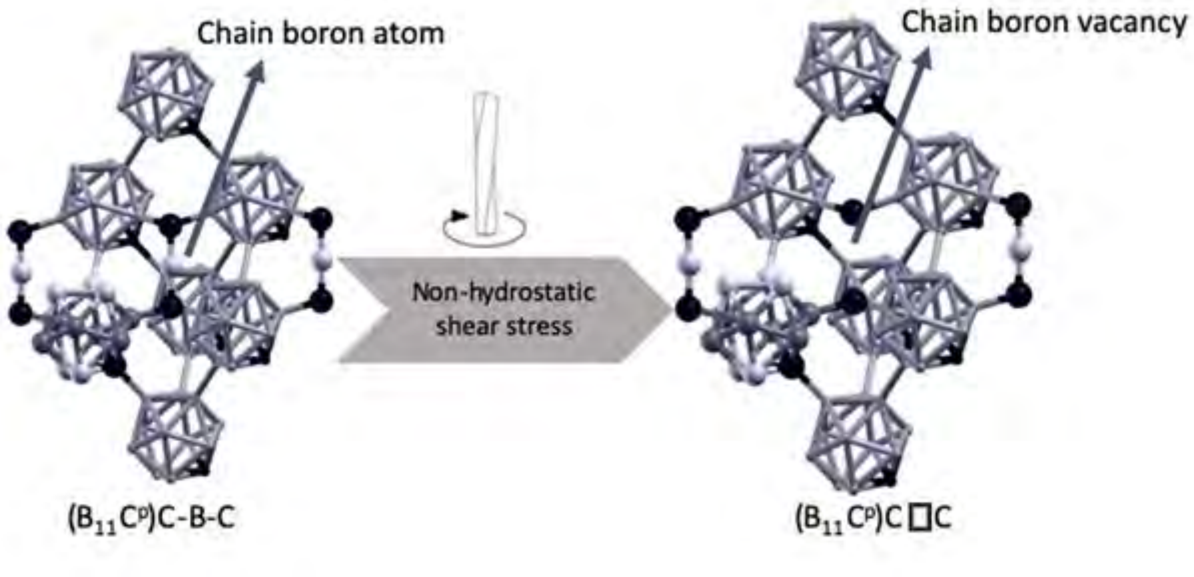
Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Boron carbide is a widely used superhard ceramic, with applications ranging from engineering tools, nuclear industry, safety armours, etc. However, it anomalously loses its high hardness when subjected to impact above 15–17 GPa. In this work, a new mechanism is proposed to explain this behaviour using combined experimental and theoretical techniques. A special recently developed rotating anvil press, the rotating tomography Paris Edinburgh cell (RoToPEC), has been used to apply torsional deformation to boron carbide under a pressure of 5 GPa at ambient temperature. Subsequent damages and point defects have been analysed at ambient pressure by energy dispersive X-ray microdiffraction at the synchrotron and by Raman spectroscopy, combined with calculations based on the density functional theory (DFT). We show that apart from the signals due to B4C, new peaks appear in both characterisation methods. The DFT calculations of atomic structures and phonon frequencies enable us to attribute most of the new peaks to boron vacancies in the intericosahedral chains of boron carbide. At the same time, we show preliminary results of the synthesis of a new high-pressure phase (above 50 GPa) of boron carbide that can resist the formation of these chain vacancies, thus potentially reinforcing boron carbide against such premature failure. This work thus provides valuable insights into high-pressure reinforcements of ceramics in general against disastrous anomalous failures.



High-pressure synthesis and study of structural and physical properties of Ba-based Ruddlesden-Popper 4d/5d transition metal oxides

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

4d/5d transition metal oxides exhibit plenty of novel physical properties due to their strong spin-orbit coupling. Here we attempt to synthesise various Ruddlesden-Popper (RP) Ban+1MnO3n+1(M=Ru, Ir) transition metal oxides by a Walker-type multi-anvil high-pressure facility and study their structural and physical properties systematically. Recent experiments have demonstrated that Ba2RuO4 in the RP series can be synthesised under 9 GPa, whereas BaRuO3 perovskite can be synthesised under 18 GPa, which suggests that the Ba2O2 rock-salt layer in the RP phase is more compressible than the BaO layer in the perovskite phase. We expect to obtain the RP series of Ban+1RunO3n+1 and Ban+1IrnO3n+1 (n=2, 3, 4...) under much lower pressures. More importantly, the physical properties of most Ban+1MnO3n+1 (M=Ru, Ir) transition metal oxides, such as Ba2RuO4 and Ba4Ir3O10, have not been fully studied. The structural characterization of the high-pressure RP series of Ban+1MnO3n+1 (M=Ru, Ir) by using X-ray diffraction, neutron diffraction and the comprehensive study of their magnetic, electrical, and specific-heat properties would help to greatly deepen our understanding of rich physical phenomena associated with the structure-property relationships.

XAS and XMCD under extreme conditions at the ODE beamline – SOLEIL Synchrotron

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The ODE beamline is one of the few X-ray absorption spectroscopy (XAS) beamlines in the world using a dispersive set-up. Instead of a step-by-step scanning of the energy, only one crystal is used, bent in such a way that it directly opens an energy range and focuses the beam. XAS measurements using this set-up present the advantages to be very fast and very stable since only a few optics are involved and no mechanical movement is required. These characteristics make the dispersive XAS technique a unique tool for investigating small samples, following kinetics and measuring data presenting very small signal to noise ratio (down to 10-5). Moreover, the ODE beam line is built on a bending magnet allowing a circular polarization of the beam, hence providing the possibility to perform X-ray Magnetic Circular Dichroism (XMCD) measurements. Thanks to its focusing optics giving rise to a spot of about 100 µm fullwidth, the dispersive set-up is also particularly well suited for high-pressure experiments in diamond anvil cells.

We will present the precise characteristics and the last improvements of the ODE beamline and illustrate its possibilities with recent high-pressure XAS and XMCD results, such as a pressure induced spin crossover transition of molecular compounds, the magnetic properties of iron hydride under pressure and the exceptionally robust magnetism and structure of SrFeO2 up to 100 GPa.

Isothermal mode Gruneisen Tensor of Cadmium across electronic and structural phase boundaries

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The fundamental properties of metals are intrinsically invoked from the lattice dynamics of the quantised movement of atoms in a periodic lattice. Measurements between 0-30 GPa and 300-700 K for ω(P, T) and V(P, T) are determined by Raman spectroscopy and X-ray diffraction techniques, respectively, to calculate the isothermal mode Grüneisen tensor of elemental cadmium with temperature. This work demonstrates that the mode Grüneisen tensor is sensitive to second order structural phase transitions and subtle electronic phase transitions.

Temperature and pressure-dependent incommensurate to commensurate on NbSe3

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Our work is focus on the crystal structure of CDW states in NbSe3 and its evolution under extremely condition. Temperature (T) and pressure (P) dependent q vectors have been performed on NbSe3 by single crystal XRD. The T dependent q1 and q2 vectors show an incommensurate to commensurate change at low T range. The comparison of evolution results of q1 with pressure at T=5 K and T=32 K, also exhibit a similar result, at lower temperature the q1 vector exhibits a suddenly decrease below 0.8 GPa, this further indicates the incommensurate to commensurate on NbSe3 below 27 K. We also performed electrical transport and magnetoresistance measurements in the same P-T range. A sign-change of magnetoresistance (positive to negative) and hall effect (n-type to p-type) at low T range are found again at 0.8 GPa, thus suggesting a dimensional cross over at this pressure. The phase diagram on NbSe3 is improved and complement. Our study on NbSe3 will help to systematise the broader class of q1D metallic system, the similarities and differences of their properties and the limits of the FS adjustment by hydrostatic pressure and uniaxial pressure.

Prediction of Above-Room-Temperature Superconductivity in Lanthanide/Actinide Extreme Superhydrides

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Achieving room-temperature superconductivity has been an enduring scientific pursuit driven by broad fundamental interest and enticing potential applications. The recent discovery of high-pressure clathrate superhydride LaH10 with superconducting critical temperatures (Tc) of 250–260 K made it tantalizingly close to realizing this long-sought goal. Here, we report a remarkable finding based on an advanced crystal structure search method of a new class of extremely hydrogen-rich clathrate superhydride MH18 (M: rare-earth/actinide atom) stoichiometric compounds stabilised at an experimentally accessible pressure of 350 GPa. These compounds are predicted to host Tc up to 330 K, which is well above room temperature. The bonding and electronic properties of these MH18 clathrate superhydrides closely resemble those of atomic metallic hydrogen, giving rise to the highest Tc hitherto found in a thermodynamically stable hydride compound. An in-depth study of these extreme superhydrides offers insights for elucidating phonon-mediated superconductivity above room temperature in hydrogen-rich and other low-Z materials.

Optical setup for fluorescence pressure measurements in piston-cylinder pressure cells with LED light source

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

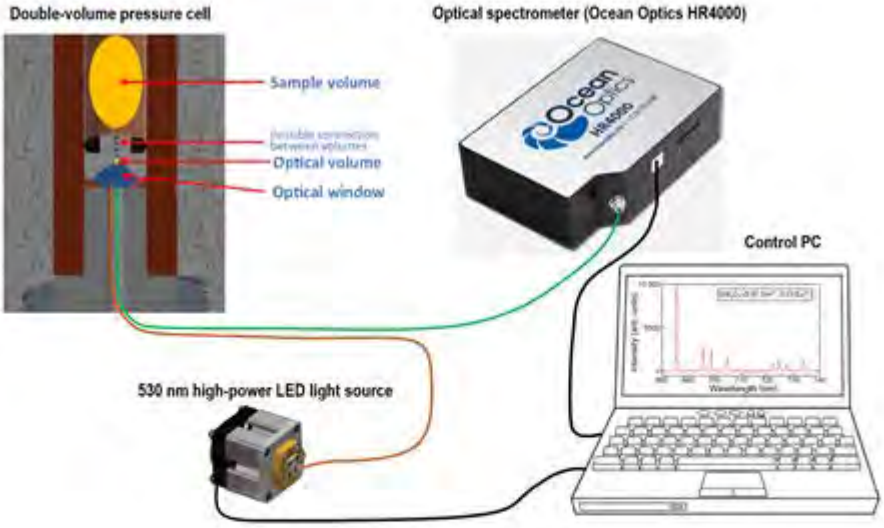
Measurement of the absolute value of the applied pressure in high-pressure muon and neutron experiments is a complicated task. It often requires the presence of a calibration material inside the sample volume, and could also cause additional time to obtain the response of the calibrant. Here we describe the use of optical calibrants for precise determination of the pressure value inside the piston-cylinder clamp cells including the setup of a separate volume seal equipped with an optical window and the corresponding fibre coupled unit for the fluorescence line determination.

Utilizing the concept of separate volumes for the sample and the optical media, a setup for conducting in situ pressure measurements is successfully tested. Pressures in both the “sample” and the “optical” volumes are proved to be the same within experimental accuracy.

The use of Strontium tetraborate $Sm^{2}+$:SrB_4O_7:($0.01Sm^{2}+$,$0.03Eu^{2}+$) as a pressure calibrant allows for a high accuracy of pressure determination by considering up to eight fluorescence lines.

As light source for the fluorescence pressure measurements commercial high-power light emitting diodes (LEDs) are used. A relatively broad light emitting spectra of single colour LEDs (in comparison with lasers) do not prevent producing narrow fluoresces lines at least for our used pressure calibrant. Strongest responses of the pressure indicators were detected for the green colour LEDs with the average wavelength λ_av= 530 nm.

The spectrum itself is recorded with a commercially available HR4000 High Resolution Spectrometer (Wavelength Range 670–771 nm).



Opportunities for high-pressure research at beamline I18 of the Diamond Light Source

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The I18 beamline at the Diamond Light Source is dedicated to microfocus X-ray spectroscopy in an energy range between 2.05 keV to 20.5 keV. The in-vacuum undulator source and an optical scheme that includes Kirkpatrick-Baez focusing mirrors, provide a flux of ~1012 ph/s in a spot of 2 µm x 2.5 µm at the sample position. The beamline offers a range of techniques including X-ray-fluorescence (XRF), X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) and is optimised for mapping and tomographic studies.

The beamline capabilities for research at high-pressure will be presented. Membrane-driven Diamond Anvil Cells are available for precise pressure tuning, together with a PACE 5000 digital pressure controller for remote operation. Mini-diamonds of 0.5 mm height can be used to reduce X-ray absorption and have allowed successful transmission XAS measurements at the low energy of the Cr K-edge (5989 eV). In-situ Raman and on-line pressure measurement are performed with an iHR320 spectrometer with 532nm laser. Additionally, large angular opening Boehler-Almax diamond anvils allow the combination of XAS with diffraction measurements.

Metallic hydroxides within the Earth’s deep-water cycle

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

In the past couple of decades, scientific research has shown that a considerable amount of Earth’s water is stored inside of the planet [1-3]. The constant exchange of fluxes between these Earth’s inner reserves and superficial water constitutes the so-called Earth’s deep-water cycle. This global process is considered to be one of the main causes of geological activity and to hugely contribute to the preservation of habitable atmospheric conditions [4]. The transportation of water from the bottom of the oceans to the Earth’s interior takes place during the subduction processes, in which hydrous minerals on the lithosphere are dragged into the Earth’s mantle. One of the most common and simplest compounds present on the oceanic hydrated crust is magnesium hydroxide, Mg(OH)2, commonly known as brucite. In this work, we study the pressure evolution of three isomorphic metallic hydroxides, M(OH)2 with M= Mg, Ni and Co, up to 40 GPa, by means of X-ray diffraction and Raman spectroscopy, as well as their decomposition mechanisms at high-pressures, using both laser and resistive heating. Our experimental results are supported by ab initio calculations.

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Evaluation of a disposal type of 6–6 frame for high-pressure experiments

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

A multianvil (MA) press has long been used to generate high-pressure in science and technology developments. Many synchrotron X-ray and neutron beam facilities operate MA presses to provide in situ measurements. In this decade, such MA presses have changed over from a traditional (one-stage) to a two-stage compression method; anvils mounted on a press compress a sample enclosed in a cell in a one-stage compression, whereas they push a set of inner anvils holding a sample cell in a two-stage compression. The inner anvils must be carefully aligned to share the centres. Here, we name this alignment 'centring.' The inner anvils and a guide bundling them, a 'frame,' constitute a second-stage assembly (SSA). In the following, we focus on an SSA consisting of six inner anvils held by six outer anvils, i.e., the so-called '6–6' assembly.

The two-stage compression method has brought about a revolutionary change; we can choose a truncated edge length of the inner anvils, depending on the desired pressure range, upon exchanging a sample, even in a single machine time. At the same time, we are more likely to lose machine time through a time-consuming preparation of an SSA, which must ensure (i) electric insulation and (ii) centring. We need (i) for a current not to bypass a sample to be heated electrically. It usually takes an hour for an expert to adjust an SSA using pieces of balsa sheets and polytetrafluoroethylene tips to meet the condition (ii).

We have cleared requirement (i) by using a plastic material with high-precision machinability comparable to metals used usually. We thus arrived at PEEK or POM as a desirable material.
To overcome the problem regarding (ii), we have developed the assembling tools, which allow even a non-expert to set up an SSA within a few minutes, thus minimizing loss of time. We demonstrated the cantering accuracy and stable operation by in situ observation conducted under high-pressure and temperature conditions [1].

Unfortunately, a PEEK or POM frame is hardly reusable after it experiences a blowout due to deterioration of the frame surfaces caused by (hot) ballistic fragments of broken inner anvils, preventing the inner anvils from moving uniformly. In contrast, a metallic frame, composed of the parts held together by cyanoacrylate, can quickly release a blowout impact by disassembling and is usually reusable if reassembled. We could recently successfully develop a disposable frame made of inexpensive plastic materials such as nylon and acrylic. Although the load-deflecting temperature of the latter materials is not as high as that of PEEK and POM, a disposable frame should be worth adopting when an experiment may end with a blowout. Thus, we can use the two types of plastic frames depending on our purpose. They now replace a metallic frame employed thus far in a 6–6 compression mode.

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Finite element analysis to extent Boehler-Almax anvils to ultra-large apertures

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Boehler-Almax diamond anvils are widely used within the high-pressure community. These diamond anvils with a 60° cone have been jointly developed by Reinhard Boehler and Koen De Hantsetters [1] and X-ray apertures up to 90 degrees have been reported [2] which, however, required large diamonds.

Here we present preliminary work to further extent the apertures by modifying the cone angle using high-strength seat materials. The anvil/seat combination is optimised by matching the cone angles on the micron level allowing forces up to at least 5000 Newton. Such developments could lead to significant improvements in high-pressure X-ray crystallography for low symmetry single crystals.

In this poster, we will present the finite element analysis for 20° cone Boehler-Almax anvils for both WC- and high-strength steel seats.

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Super-hydration and reduction of manganese oxide minerals at shallow terrestrial depths

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Manganese oxides are ubiquitous marine minerals which are redox sensitive. As major components of manganese nodules found on the ocean floor, birnessite and buserite have been known to be two distinct water-containing minerals with manganese octahedral interlayer separations of ~7 Å and ~10 Å, respectively. We show here that buserite is a super-hydrated birnessite formed near 5 km depth conditions. As one of the most hydrous minerals containing ca. 34.5 wt. % water, super-hydrated birnessite, i.e., buserite, remains stable up to ca. 70 km depth conditions, where it transforms into manganite by releasing ca. 24.3 wt. % water. Subsequent transformations to hausmannite and pyrochroite occur near 100 km and 120 km depths, respectively, concomitant with a progressive reduction of Mn4+ to Mn2+. Our work forwards an abiotic geochemical cycle of manganese minerals in subduction and/or other aqueous terrestrial environments, with implications for water storage and cycling, and the redox capacity of the region.

Superconductivity in Zirconium Polyhydrides with Tc above 70 K

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Hydrogen is expected to be a metal at sufficient high-pressures while it can also be a high temperature superconductor based on BCS theory because of its high Debye temperature arising from the light mass of hydrogen. Since it is very challenging to reach the pressure for the hydrogen metallization, the ‘‘chemically precompressed” hydrogen-rich compounds were in turn proposed to realise high temperature superconductivity at lower pressures. The experimental discovery of high temperature superconductivity in SH3 has greatly stimulated efforts to explore new hydrogen-rich superconductors, such as in rare earth polyhydrides. Here we report the discovery of superconductivity with Tc about 70 K in zirconium polyhydrides, the record high Tc so far for 4d transition metal hydrides. The new materials are synthesised above 200 GPa and 2000 K by using diamond anvil cell (DAC) combined with laser heating.

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Reaction between the feldspars and water in the Earth’s transition zone

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Water transport and cycle to the deep Earth are important in the dynamics and evolution of the Earth as water affects the phase transition kinetics as well as the viscosity of the mantle by the process called hydrolytic weakening. Delivery of water to the deep Earth has operated since the initiation of modern-style subduction processes. Phase transitions of subducting minerals to dense hydrous silicate minerals during subduction is one of the key processes to transport water into the deep interior. And one of the under-explored host candidate minerals for water in the Earth’s deep interior is hollandite-type minerals which are the transformation products from subducting feldspars and known to be stable down to the core-mantle boundary conditions. Therefore, studies on the physico-chemical properties of feldspars will extend the understandings of the Earth’s volatile cycles and dynamic processes.

Feldspars, a family of framework silicate minerals containing Na, K, and Ca, are one of the major constituents in the crust. When feldspars in the oceanic crust subduct along dry geothermal gradients, they have been known to breakdown into an assemblage containing a hollandite structure in the conditions of the mantle transition zone. In recent years, the presence of water in the mantle transition zone has been suggested by seismic studies and high-pressure experiments. The hollandite structure, possibly formed under conditions of the transition zone, is characterised by its high affinity for large cations with low valence due to its unique 2 x 2 octahedral tunnel structure where it can store and transport water and large cations such as 40K radioactive heat source which could affect the thermal evolution of the Earth.

In recent years, we have reported the formation of smectite and moganite assemblage as hydration breakdown of the albite in the intermediate depth range between 90 and 135 km, under wet and cold subduction conditions. This calls follow-up investigation at deeper depths to investigate whether hollandite-type feldspars can be formed under the similar, wet conditions, as a possible host for water and alkali cations in the lower mantle. In our LH-DAC experiments, we observed the formation of the hollandite-type structure from feldspars and the expansion of its unit cell volume when it forms under wet transition zone conditions compared to a dry analogue. This could be an indication of hydration in the tunnel-structured hollandite. This result could provide important data to understand the cycle and transportation of water and heat in the deep mantle and possibly the core mantle boundary.

Hydration breakdown of serpentines along cold core geotherm

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The stability of serpentine minerals are of prime importance to typify subduction zone lithology and related geochemical cycling. In this study, we conducted in-situ high-pressure and temperature X-ray powder diffraction experiments on lizardite and antigorite (Mg3Si2O5(OH)4), the two representative serpentine minerals, using a resistive-heated diamond anvil cell (RH-DAC) under two contrasting geothermal paths of a water-saturated cold subduction zone. Along cold moho subduction geotherm with ~2 °C/km gradient, lizardite is decomposed to the previously known phase assemblage of Phase E (Mg2SiO2(OH)4) and clinoenstatite (MgSiO3) above ca. 8 GPa and 670 °C, equivalent to ca. 240 km depth. This corresponds to a typical dehydration reaction to release water by ~1 wt. % from subducting lizardite into the region. In contrast, along cold core geotherm of ~1.6 °C/km gradient, lizardite undergoes previously unknown breakdown to form the 3.65 Å phase (MgSi(OH)6) and phase E assembly above ca. 9.5 GPa and 500 °C, equivalent to ca. 285 km depth. This is a new type of hydration reaction to incorporate more water by net ~22.6 wt. % from the surrounding subduction fluid to form the DHMS (dense hydrous magnesium silicate) assemblage. In the case of antigorite, such a hydration breakdown is confirmed at marginally deeper depth at ca. 300 km. The newly observed hydration breakdown into the DHMS assemblage manifests a new type of metamorphic reaction specific for the cold core condition, which, coupled to the dehydration reaction along the cold moho geotherm, demonstrates lateral variations in water transport capacity of a subducting slab. This could also be related to the earthquake distribution into double seismic zone (DSZ), which marks seismic characteristics for a cold subducting slab.

High-pressure study of the van-der-Waals ferromagnet CrBr3

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Two-dimensional van-der-Waals (vdW) magnetic materials have recently become the subject of an intensive research owing mainly to their promising application potential in spintronic and electronic devices. In Particular, the family of layered van-der-Waals (vdW) trihalides TX_3 sparked much attention due to the first known example of layered material retaining stable ferromagnetic order in monolayer limit in case of the CrI_3.

Here we report a results of comprehensive investigation of layered vdW ferromagnet $CrBr_3$. We have studied the effects of hydrostatic pressures by means of SQUID magnetometry (up to 9GPa), electrical resistivity (up to ~ 30GPa) and Raman scattering (up to ~ 10 GPa). In addition, we have measured the magnetization of $CrBr_3$ under uniaxial compression in direction perpendicular to the van-der-Waals layers (up to ~0.6GPa).

Magnetization measured under hydrostatic pressures in DAC (Daphne 7575 PTM) shows gradual decrease of Curie Temperature from its ambient pressure value TC = 31.5 to being completely suppressed around ~8.5 GPa. This is well corresponding to the recent work which suggested the suppression of magnetic ordering by extrapolating HP neutron diffraction data measured < 3GPa and further corroborated existence of some electronic transition by sudden change in EOS at the same pressure by X-ray diffraction [1]. On the other hand, the measurement of magnetization under uniaxial compression showed almost negligible effect on both, value of saturated moment and ordering temperature TC, suggesting the interplanar interactions play only minor role in the magnetism of the compound.

In the room temperature Raman measurement, we have seen the splitting of Ag_1 and Ag_2 peaks, with onset at pressures ~2.5GPa. This is most probably related to the structural change from trigonal R-3 to monoclinic C2/m structure, which has been reported within entire TX_3 series (T=V,Cr, X=I, Br,Cl). Previous works reported the structural transition to appear at 450K at ambient pressure in case of $CrBr_3$ [2], pointing to rather high value -60K/GPa. With a dose of imagination, extrapolating to low temperatures points to similar pressures where the magnetic ordering disappeared.

Electrical resistivity has been measured in DAC, using solid pressure medium with thin golden contacts pressed directly onto the sample surface. In the entire range of measured pressures, the resistivity had an insulating/semiconducting character, i.e. rapidly increasing with decreasing temperature. Both, the overall resistivity and the value of the activation energy deduced from the temperature dependence, decrease almost linearly with pressure up to 30 GPa. Extrapolating the data, critical pressure for inducing the possible metallization can be roughly estimated as ~ 35 GPa.

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Three-wall piston-cylinder type pressure cell for muon-spin rotation/relaxation experiments

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

A three-wall piston-cylinder type hybrid pressure cell was successfully designed, produced and commissioned. The cell is made out of NiCrAl and MP35N nonmagnetic alloys with the design and dimensions specifically adapted for muon-spin rotation/relaxation measurements. For two identical three-wall piston-cylinder high-pressure cell designs, the elastic and plastic deformations were modelled using FEA to find the cell expansion during pressurisation and the resulting plastic deformation at the bore. The simulation and experimental results were very similar, where the simulation results were conservative in both cases by only 0.01 mm for cell expansion and between 0.01-0.02 mm for bore diametral plastic deformation.

The present design of the three-wall piston-cylinder pressure cell with an outer MP35N sleeve and two inner NiCrAl cylinders allows safely reach pressures up to ~3.3 GPa at room temperature (~ 3.0 GPa at low temperatures) without irreversible plastic deformation of the pressure cell walls. Test muon-spin rotation/relaxation experiments were performed on the elemental Indium sample.

High-Pressure Study of Electrical Transport Properties of VBr3 and CrBr3: Possible Pressure Induced Metallization

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

In recent years, two-dimensional van-der-Waals (vdW) magnetic materials have become the subject of an intensive research mainly because of their promising application potential in spintronic devices. In Particular, the family of layered vdW trihalides TX_3 attracted much attention due to the first known example of material retaining stable ferromagnetic order in monolayer limit in case of the CrI3.

In the presented work, we have focused on pressure evolution of electronic properties of VBr3 and CrBr3 samples of the TX3 family. At ambient pressure, CrBr3 is a ferromagnet with Curie temperature TC = 31.5 K, while VBr3 is an antiferromagnet with Neel temperature TN = 26.5K. Electronically, the CrBr3 is known to be Mott insulator, while the VBr3 has been recently suggested to be on the border between Mott and the charge-transfer one [1]. Previous optical measurements at ambient pressure estimated the energy gap values to be 1.68-2.1 eV and ~1.1 eV for CrBr3 and VBr3, respectively [1,2]. In case of CrBr3, the value of the gap is debated due to some controversial results, e.g. with a smaller ~0.57eV gap recently reported for the CrBr3 by scanning tunnelling microscopy current analysis [3]. Due to the volatile nature of the samples and very low conductivity (e.g. CrBr3 ~ 109ohm.cm-1 at 300K), these measurements are extremely challenging. Only one ambient pressure work showing the resistivity of bulk CrBr3 crystals [4] and recent high-pressure work demonstrating the metallization of related CrI3 [5] has been published.

Here, we have measured the electric transport properties of VBr3 and CrBr3 single crystals under high-pressures up to 13GPa and 25GPa, respectively. Our data show gradual decrease of the overall sample resistivity as well as a gradual change in character of their temperature dependence. With a detailed analysis of the R(T) data, we estimated the pressure evolution of energy gap (Eg). For both samples, deduced values of Eg shows linear decrease with applied pressure. By extrapolating our data to higher pressures, the insulator-to metal transition can be expected around ~23GPa for VBr3 and ~ 35GPa for CrBr3. Moreover, there is a qualitative difference in scaling ln(R/R0) vs (1/T)b, which shows b=1 dependence expected of simple insulator in the case of VBr3, while the data for the CrBr3 show b=1/2 dependence characteristic for variable-range hopping. This is consistent with the recently reported band calculations and STM current spectroscopy results [3].

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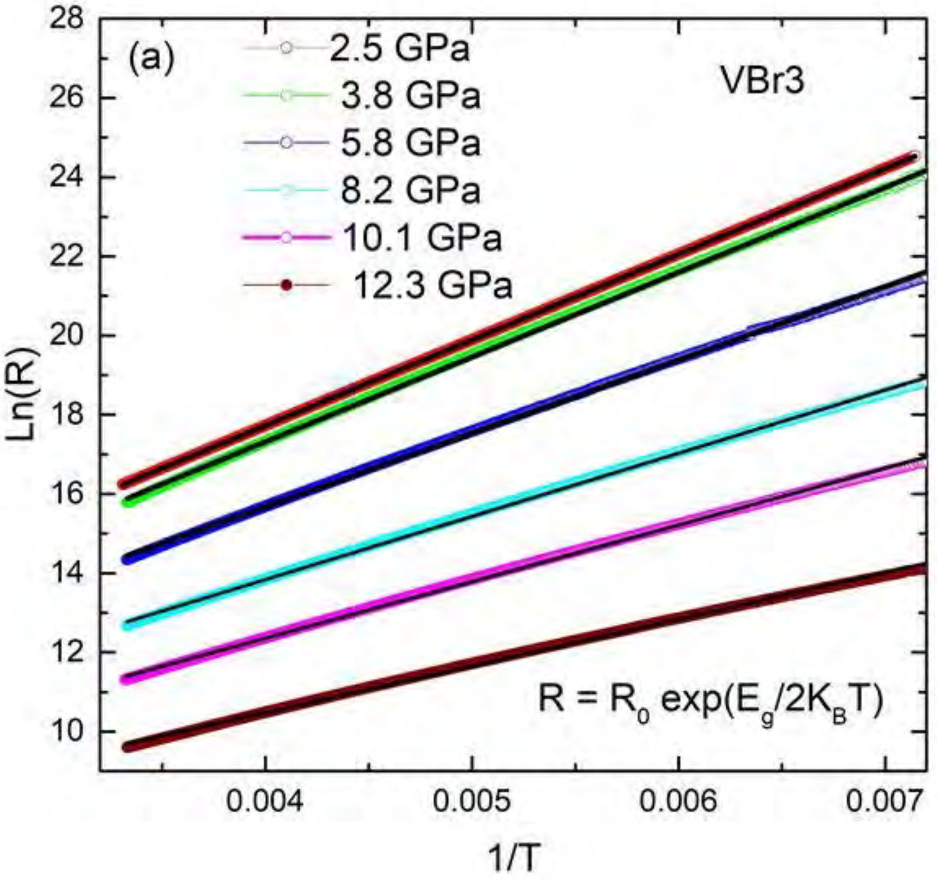
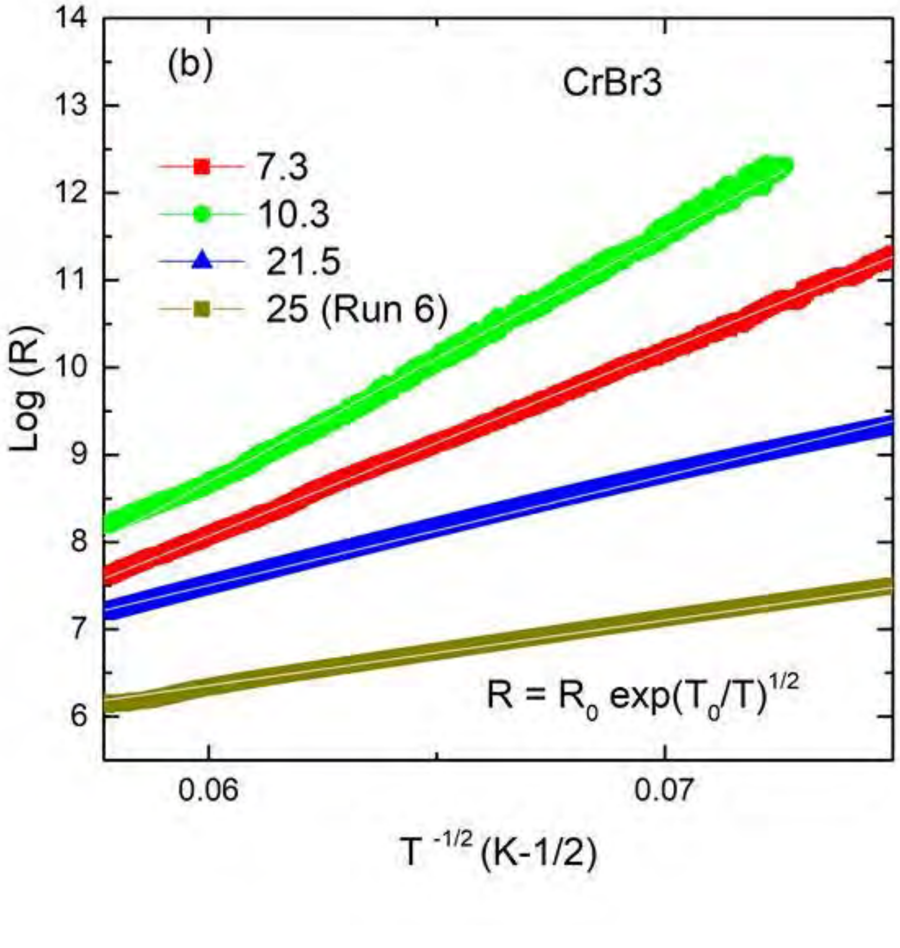

(a)
VBr3
2.5 GPa
3.8 GPa
5.8 GPa
8.2 GPa
10.1 GPa
12.3 GPa
$R = R_0 \exp(E_g/2K_BT)$
Ln(R)
28
26
24
22
20
18
16
14
12
10
0.004
0.005
0.006
0.007
1/T


(b)
CrBr3
7.3
10.3
21.5
25 (Run 6)
$R = R_0 \exp(T_0/T)^{1/2}$
Log (R)
14
13
12
11
10
9
8
7
6
0.06
0.07
$T^{-1/2}$ (K-1/2)

High field neutron study and complex magnetic structures of the NTO-type solid solution Ni2-xCoxScSbO6

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

ABO_3 oxides have proven to accommodate a wide variety of chemical compositions, to crystallise with several structures in competition and to develop diverse physical properties. Multiferroic (e.g. $LiNbO_3$-type $MnTiO_3$-II) [1,2], where magnetism and ferroelectricity coexist, have been intensely investigated and presented a wide range of potential applications such as transducers, actuators and sensors [3]. Comprehensive studies of structural and magnetic properties of the cation-ordered Ni_3TeO_6 (NTO) show an acentric rhombohedral space group R3 crystal structure and novel multiferroic properties [4]. Recently an NTO-type material Co_2ScSbO_6 was reported and found to have a helimagnetic spin order with propagation vector k = [0 0 kz] below TC = 60 K [5].

We recently investigated the solid solutions of Ni_2-xCoxScSbO_6 using neutron powder diffraction under multiple magnetic fields. Studies on the temperature dependence of the magnetisation and magnetic hysteresis indicated the occurrence of antiferromagetic ordering below TC = 65 K. Metamagnetic transition was observed in x < 0.5 and the field neutron diffraction data confirms the presence of intermediated magnetic phases. Here we will report the magnetic structures changing from a simple incommensurate helical magnetic structure to a mixture of commensurate and incommensurate magnetic phases where a third magnetic sublattice active under critical magnetic field. The presence of electrical polarisation and spontaneous magnetisations offers possibilities for multiferroic properties.

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Chemical interactions of iron and methane at extreme conditions

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Compounds of planetary ices, specifically methane, and mixtures of methane and water, methane hydrates (MH), are believed to be present in a variety of planetary bodies, both within and beyond our Solar system. The presence of methane on exoplanets could be a potential indicator of the presence of life and can impact assessments of planetary habitability [1,2]. Additionally, methane and methane hydrates are considered to constitute the interiors of (exo)planets, and therefore, to be significant factors in the (exo)planetary evolution [3].

Despite this importance, the chemical behaviour of CH_4 and CH_4/H_2O in the presence of other elements at the high-pressure and high-temperature (HP-HT) conditions relevant for planetary bodies is poorly known. Possible chemical interactions between methane and methane hydrates with iron-rich mantle silicates and iron-alloyed core materials might cause the formation of yet unknown compounds within the Fe-C-O-H system. An improved understanding of this system is thus crucial for the modeling of the interiors, dynamics, and evolution of ice planets and exoplanets.

We investigated the chemical reactivity between iron and methane (as a model system) in a series of experiments performed at conditions from 10 up to 60 GPa and 1200(100)-3500(100) K in laser-heated Diamond Anvil Cells. Using in situ synchrotron single-crystal and powder X-ray diffraction (SC-XRD), we observed that at these conditions, relevant for the (exo)planetary interiors, chemical interactions between methane and iron lead to the formation of a series of iron carbides (such as Fe_7C_3 and Fe_3C) and iron hydrides (dhcp-FeH, fcc-FeHx). Our results imply that the formation of such compounds might occur in the interiors of (exo)planets, therefore, adding possible necessity for refining existing (exo)planetary models.

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Second Order Phase Transition and Stabilizing CH-H and CH-S Interactions in Naphthyl End-Capped Bithiophene at 3.5 GPa

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The organic semiconducting molecule, 5,5′-bis(naphth-2-yl)-2,2′-bithiophene (NaT2) is used in thin film applications such as photosensor or phototransistors. In pi-conjugated and related systems, charge transportation and other properties have been shown to depend on such factors as: intermolecular separation, relative displacements between molecules, and cluster and crystallite size. [1,2] Pressure can be used as a clean tool to modify the molecular displacements and hence electronic properties without chemical addition.

Here we report the high-pressure structures of NaT2 to 7.6 GPa and characterise its phase behaviour by single-crystal X-ray diffraction, photoluminescence, Raman spectroscopy and empirical energy calculations. NaT2 retains its ambient pressure structure (P21/c) on compression but undergoes a subtle second-order phase transition at 3.5 GPa. This is characterised by the formation of new S···H interconnects and a change in the compression pathway along the long molecular chain direction. Finally, NaT2 undergoes a bandgap closure on compression, manifested as a yellow to red colour change, that is fully reversible on decompression.

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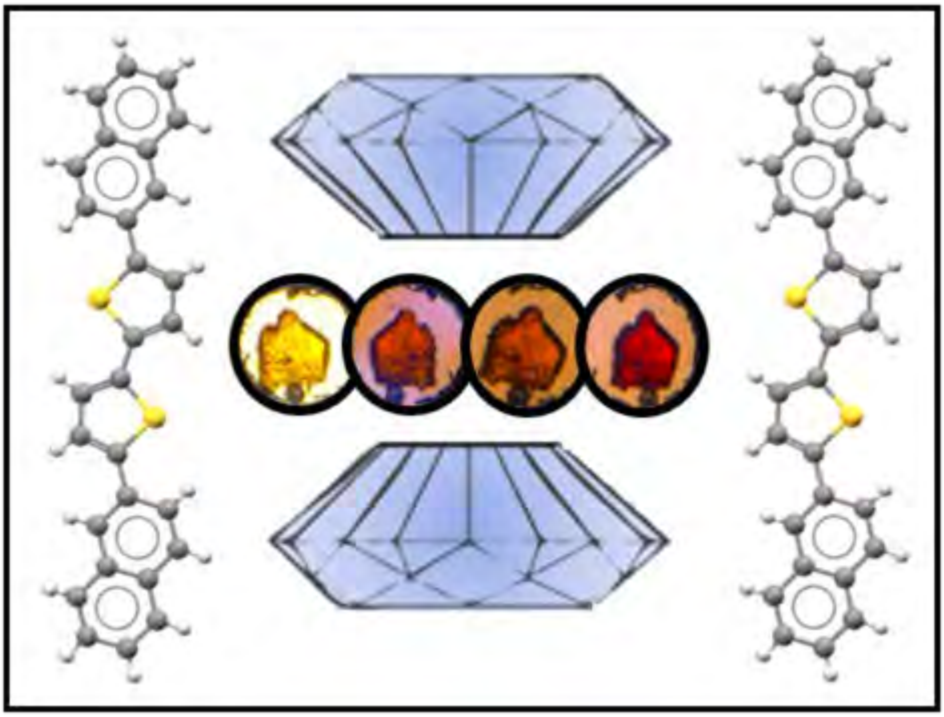

Are you using the wrong EoS in Dioptas?

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

DIOPTAS [1] is a popular and comprehensive python-based software package that enables users to integrate and analyse two-dimensional diffraction data collected on a wide range of detector geometries. The software includes a parametric framework for representing a material's crystallographic and volumetric behaviour across a broad range of pressures and temperatures allowing for the comparison of calculated Bragg peak positions against experimental data in real-time at the click of a button.

In this work, we have developed a simple, least-squares minimisation procedure for attaining DIOPTAS-specific thermal equation of state (EoS) parameters from existing models or pressure-volume-temperature data in the literature such that the EoS is best represented within DIOPTAS. We provide a .xlsx file for readers to perform refinements for any literature EoS as well as the .jcpds files for a number of standard reference materials and pressure media frequently used in high-pressure experiments including Al, Au, Cu, Mo, Nb, Pt, Ta, hcp-Fe, MgO, NaCl-B1, NaCl-B2, KCl-B2, and Ne [2]. The .jcpds and .xlsx files are available from https://gitlab.com/jdmchardy/dioptas-jcpds

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High-pressure Raman study of ibuprofen

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Ibuprofen is a very common non-steroidal anti-inflammatory active pharmaceutical ingredient, used in a plethora of over-the-counter products, acting as a cyclo-oxygenase inhibitor and leading to significant reduction of prostaglandins, key factors in the production of pain, fever and inflammation [1]. The knowledge of the phases of a substance of pharmaceutical interest is essential for its use, while Raman spectroscopy is a versatile technique to investigate the structural stability and phase transitions of materials. In this work, commercially available crystalline racemic ibuprofen was subjected to pressures up to ~6 GPa in a diamond anvil cell using glycerol as pressure transmitting medium (solidification at 5.5 GPa [2]) and probed by Raman spectroscopy by means of a LabRAM HR (HORIBA) single stage spectrometer with a spectral resolution of ~3.5 cm-1. Raman spectra were excited by the laser emission of a 514 nm diode-pumped solid-state laser at a power of 1 mW, delivered on the sample by a 50x (N.A. 0.45) long working distance objective.

The Raman spectrum of ibuprofen is very rich, owing to the low symmetry of the molecule and its monoclinic crystal structure, P21/c (phase I) [3]. In the low frequency spectral region (<250 cm-1), the spectrum is populated by the intermolecular (lattice) modes and the low frequency intramolecular modes, while the intermediate frequency region (250-1700 cm-1) contains the backbone and aromatic vibrational modes, and the high frequency region (2700-3300 cm-1) is dominated by the C-H stretching vibrations. Pressure application causes the shift of the observed Raman peaks to higher frequencies, as well as changes in their relative intensities. The pressure dependence of the frequencies of all the Raman peaks is quasi-linear up to ~2 GPa. However, for higher pressures, significant changes occur with respect to the Raman spectral profile and the pressure slopes of the peak frequencies, being more evident in the intermolecular and the C-H stretching spectral regions. The observed changes are compatible with the pressure-induced isosymmetric structural transformation of ibuprofen previously reported by means of X-ray diffraction [3]. It appears that this transformation is accompanied by rather small modifications in the molecular conformation. The high-pressure structure is stable at least up to 6 GPa, while the observed transformation is fully reversible upon pressure release.

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The high-pressure behaviour of $Nd(XeF_2)_3(TaF_6)_3$

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

High pressure single crystal X-ray diffraction is a well-established technique in chemical crystallography, used to examine the behaviour and response of compounds to pressures in the 10s of GPa range. This enables researchers to study the phase behaviour of materials, their compression dynamics and structural phase changes, and thus infer information about the intermolecular interactions. This technique has been widely applied to a range of compounds including molecular organics,[1] inorganic coordination compounds.[2]

The first compounds containing noble gases were discovered in 1962.[3] Since then, the chemistry of xenon, particularly in combination with fluorine, has been extensively developed.[4] The crystal structures of xenon fluorides exhibit many short intermolecular contacts as a result of the high Lewis acidity of the Xe atom. The response of these contacts to pressure can potentially lead to phase transitions, conversion of long contacts into primary bonds, or other structural changes. Research in this area has thus far been very limited, with only high-pressure powder diffraction studies of XeF_2 having been reported.[5] Our current work seeks to address this and investigates the behaviour of more complex XeF_2 coordination compounds under pressure.

The compound $Nd(XeF_2)_3(TaF_6)_3$ (1) has provided unique insight into the phase and structural characteristics of XeF_2 coordination compounds at high-pressure. Compression studies of 1 have been performed in a range of hydrostatic media, including inert gases and halocarbon oil, up to pressures of 5.5 GPa, with distinct differences in behaviour depending upon the choice of hydrostatic medium. Compression in halocarbon oil leads to a first order phase transition at 2.7 GPa characterised by collapse of channels formed along the c-axis. Unusually, one Xe site in the high-pressure phase exhibits fewer direct and long-range Xe-F interactions than at ambient pressure.

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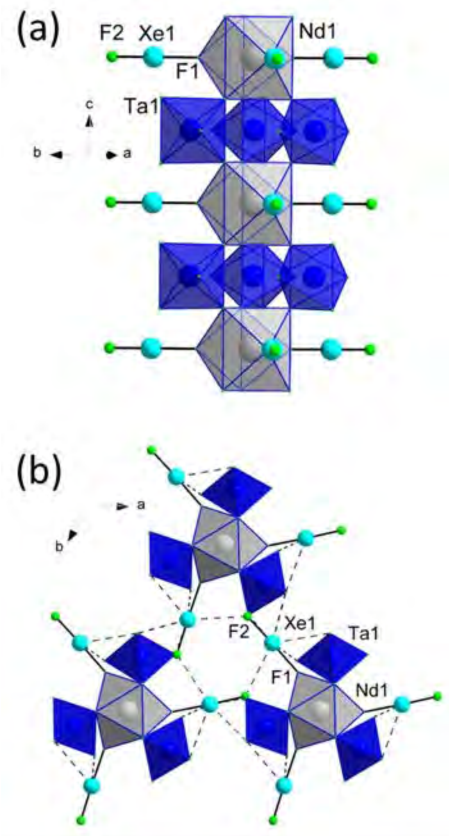

Figure 1: The crystal structure of $Nd(XeF_2)_3(TaF_6)_3$ [**1**] at ambient pressure and 100 K (R_1 = 1.45%). (a) Chains formed along the *c* axis. (b) Xe...F contacts connecting chains.

High-pressure crystal structure study of non-superconducting $Ln_4Ni_3O_8$ and $(Ln_{1-x}A_x)NiO_2$ layered bulk nickelates (Ln = La, Pr, Nd ; A = Sr, Ca)

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

The discovery of superconductivity below Tc ~ 15 K in thin films epitaxially grown on $SrTiO_3$ (STO) of doped infinite layer $(Nd_{1-x}Sr_x)NiO_2$ (Nd,Sr) nickelate mid-2019 has generated an enormous interest in the condensed matter community because of their great similitude with cuprates [1]. Then, superconductivity was also found in films with other rare earth elements and Ca^{2+} instead of Sr^{2+} as dopant, i.e. in La,Sr (or Ca) [2,3] and Pr,Sr [4] based systems. Very recently superconductivity was also reported for thin films of $Pr_{1-x}Sr_xNiO_2$, not grown on STO, but on $(LaAlO_3)_{0.3}(Sr_2AlTaO_6)_{0.7}$ (LSAT) substrates [5,6] with maximal Tc up to 20 K. Nevertheless, the direct impact of the in plane compressive strain, which is larger in the case of LSAT compared to STO, remains unclear. On the other hand, pressurised $(Pr_{0.82}Sr_{0.18})NiO_2$ thin film using Daphne oil or glycerol as the liquid pressure transmitting medium shows a continuous Tc enhancement over 30 K up to 12 GPa [7] which is not explained up to now.

In our laboratories, we have synthesised and characterised at ambient pressure good quality polycrystalline samples of the “trilayer” $Nd_4Ni_3O_8$ and $Pr_4Ni_3O_8$ nickelates, but also of the infinite layer $LaNiO_2$, $(Pr_{1-x}Sr_x)NiO_2$, $(Nd_{1}\ xSr_x)NiO_2$, and the $(Nd_{1}\ xCa_x)NiO_2$ series doped with Sr or Ca up to x(Sr) = 0.3 and x(Ca) = 0.5 respectively. So far, no superconductivity was found in any of these compounds but a spin-glass like and insulating behaviour at low temperature are observed.

Then, we have studied the crystal structure changes of $Pr_4Ni_3O_8$, $(Nd_{1-x}Sr_x)NiO_2$, (with x= 0, 0.07), $(Nd_{0.8}Ca_{0.2})NiO_2$ and $(Pr_{0.8}Sr_{0.2})NiO_2$ under high-pressure by synchrotron X-ray diffraction. We do not observe any structural transition in these compounds up to ~ 20 GPa whereas in $La_4Ni_3O_8$ a reconstruction of LaO layers have been evidenced at ~21 GPa [8]. The compressibilities of a- and c-axis were calculated, and bulk modulus were determined to be around 154 GPa and in the range 133-147 GPa for trilayer and infinite layer systems respectively.

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MetalJet X-ray sources for Experiments at Non-ambient Pressures and Temperatures

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

The interest of exploring new materials at more extreme condition is becoming increasingly important both for fundamental research as well as for application in e.g. superconductors and hydrogen storage. An important tool to characterise and understand these materials is by applying High-pressure X-ray diffraction HPXRD. This application relies on high energy X-rays to achieve good transmission through the Diamond anvil cell (DAC) and in order to capture large part of the reciprocal space. In addition, with smaller crystals higher pressures can be achieved at the expense of diffraction intensity. For this reason, more advanced application of this technology has so far been limited to a rather limited number of synchrotron beam lines. To aid the scientists and to accelerate research we have demonstrated unprecedented high energy beams suitable for high-pressure application utilizing the latest high power MetalJet microfocus Xray source. In this case the E1+ using I2 Indium alloy.
The X-ray source was coupled to a special high grade Montel optic with slits.

The main beam characteristics were as follows:

– Monochromatic 24keV (Indium Kα)
– Down to 10µm beam size (at sample position)
– 2-15 mRad divergence (slit controlled)
– Flux approximately 1e7 photons /sec. for 10 µm beam.

Here, we demonstrate the setup and provide first glimpse of HPXRD results possible from this unique setup. We will also show how MetalJet X-ray sources were used to perform non-ambient temperature experiments using scattering and diffraction techniques by our users to understand material transformations.

Record High Tc Element Superconductivity in Titanium at High Pressure

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

It is challenging to search for high Tc superconductivity (SC) in transition metal elements wherein d electrons are usually not favoured by conventional BCS theory. Here we report experimental discovery of surprising SC up to 310 GPa with Tc above 20 K in wide pressure range from 108 GPa to 240 GPa in titanium. The maximum Tconset above 26.2 K and zero resistance Tczero of 21 K are record high values hitherto achieved among element superconductors. The Hc2(0) is estimated to be ~32 Tesla with coherence length 32 Å. The results show strong s-d transfer and d band dominance, indicating correlation driven contributions to high Tc SC in dense titanium. This finding is in sharp contrast to the theoretical predications based on pristine electron-phonon coupling scenario. The study opens a fresh promising avenue for rational design and discovery of high Tc superconductors among simple materials via pressure tuned unconventional mechanism.

Superconductivity Observed in Tantalum Polyhydride at High-Pressure

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

We report experimental discovery of tantalum polyhydride superconductor. It was synthesised under high-pressure and high temperature conditions using diamond anvil cell combined with in-situ high-pressure laser heating techniques. The superconductivity was investigated via resistance measurements at pressures. The highest superconducting transition temperature Tc was found to be ~ 30 K at 197 GPa in the sample that was synthesised at the same pressure with ~ 2000 K heating. The transitions are shifted to low temperature upon applying magnetic fields that support the superconductivity nature. Our high-pressure in-situ XRD results suggest that the superconductivity may arise from I-43d phase of TaH3. It is, for the first time to our best knowledge, experimental realization of superconducting hydrides for the VB group of transition metals.

Analysis of ammonia-methane mixtures under high-pressure and high-temperature

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Ammonia and methane are some of the major constituents of the interiors of the giant gas planets of the Solar System. In Uranus and Neptune, their presence in the form of superionic phases could be the source of their unusual magnetic field [1]. The study of these two compounds is essential to the understanding of the properties of the planets' interiors. Investigations at planetary relevant conditions have been carried out on the phase diagrams of pure ammonia [2] and methane [3,4,5]. Mixtures of ammonia, methane and water were also previously studied at room temperature and at pressures of up to 13.4 GPa, showing the formation of methane hydrate II and III, ammonia hemihydrate-II and ice VI [6,7]. However, it is still unknown whether ammonia and methane stay in separate phases, mix or react when subjected to high-pressures and temperatures.

Here we present the first experimental report on ammonia and methane mixtures. We performed experiments on mixtures of ammonia and methane loaded in a diamond anvil cell, using Raman spectroscopy and X-ray diffraction as our main diagnostic tools. Laser heating of the sample and synchrotron angle-dispersive powder X-ray diffraction were employed.

Evidence of the formation of new compounds were observed at room temperature and at a pressure around 30 GPa. It became more noticeable following the laser heating and pressure increase.

Acknowledgements

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Magnetic properties of layered van-der-Waals ferromagnets VI3 and CrI3 under high-pressures

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

VI_3 and CrI_3 belong to two-dimensional van-der-Waals (vdW) materials. In these compounds, high hydrostatic pressure represents a powerful tool for tuning the dimensionality of the system from the original 2D character of the layered weekly bonded structure to more 3D-like character by compressing the vdW layers together. At room temperature, VI_3 crystallises in the trigonal structure [1]. Upon cooling, it undergoes a structural phase transition and below TC = 50 K hard ferromagnetic phase with strong anisotropy is established. CrI_3 is a semiconductor and exhibits a transition to an anisotropic 3D-Ising ferromagnetic state with easy axis perpendicular to the vdW layers. Mild increase of the Curie temperature from its ambient pressure value TC=61K has been reported in pressures up to 1 GPa [2].

In the presented work, we have studied the magnetic properties of VI_3 and CrI_3 in extended pressure range by means of DAC for SQUID magnetometry up to 10 GPa [3]. Experiments were carried out using MPMS (Quantum design) with a ruby used to determine the pressure. AC susceptibility was measured in piston cylinder cells up to 3 GPa with use of PPMS (Quantum design). Liquid hydrostatic medium Daphne 7373 has been used. For VI_3, the AC susceptibility data clearly reveals the ferromagnetic transition at ~ 50 K. In addition, three less pronounced peaks are observed between ~ 52 K and 60 K. The anomalies seem to be almost unaffected by increasing pressure up to ~ 0.8 GPa. Above this pressure value, we observed the peaks merging into one and simultaneously TC increases abruptly by 20% in 1.2 GPa. For higher pressures up to 3.5 GPa, TC increases linearly, followed by another sharp increase, ultimately reaching nearly 100 K at 7.3 GPa. On the other hand, we have observed a significantly different pressure effect in CrI3 compound, contradicting the results reported in [4]. We observed no significant increase of TC in low pressures up to 0.9 GPa. The value of TC does not change considerably in pressures up to ~ 1.5 GPa. At higher pressures TC starts to decrease. This linear decreasing tendency is observed up to the highest applied pressure of 6 GPa.

This different character of the pressure dependence of the ac susceptibility in the VI_3 and CrI_3 compounds is tentatively attributed to different evolution of dimensionality of ferromagnetic order, respectively.

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Composition and pressure effects on thermal conductivity of terrestrial planetary cores: Canyon Diablo iron meteorite as a natural analogue

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

Synchrotron-based X-ray diffraction of Canyon Diablo iron meteorite at simultaneously high-pressures and temperatures reveals evidence for the face-centred cubic γ high-temperature phase being present above 1500 K (and up to ~2500 K) at 60 GPa, and the hexagonal close-packed ε high-pressure phase from room temperature up to ~1700 K at 60 GPa, and up to ~1500 K at 32 GPa. These results are consistent with previous experimental reports on the high P–T crystalline phase transitions in pure iron, indicating that impurities and minor elements in natural samples may not significantly affect the crystal-structural phase-transition conditions. The thermal and electric properties of iron and iron alloys have been widely studied at high-pressures and temperatures to better comprehend the thermal evolution of planetary cores (Mattasov 1977, Bi et al. 2002, Gomi et al. 2013, Konopkova et al. 2016, Ohta et al. 2016, Hasegawa et al. 2020, Zhang et al. 2020). However, iron meteorites are considered to be remnants of the Solar System's terrestrial core-forming material and to date their thermal properties have not been studied at high-pressures. Using time-domain thermoreflectance (TDTR) we directly measure the thermal conductivity of Canyon Diablo iron meteorite. This method has been demonstrated on pure iron and iron-silicon alloys at room temperature (Hsieh et al. 2020), and we find consistent results for the Canyon Diablo meteorite. Understanding the effects of alloying is critical for determining electron- and phonon-scattering mechanisms that control heat transport across terrestrial planetary cores.

High-Pressure EOS and Phase Transition in the Pb-Sb Alloy System

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

While much is known about the high-pressure phases and EOS for elements, various alloy systems are less characterised under pressure.

This work fills in the EOS parameters of different phases in the Pb-Sb alloy systems under compression, which can be used to parametrise thermodynamic models that describe alloy phase diagrams under pressure.

Pressure range containing phase transitions of both elements: Pb: FCCHCP Sb: A7 (rhombohedral)Incommensurate (interpenetrating sublattices).

The samples: heterogeneous mixture of Sb2.4at.%Pb ; Pb6.7at.%Sb (by SEM analysis).

XRD-DAC mapping of PbSb to 20 GPa.

Superconductivity in Novel Carbon Nanostructures

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Density functional theory (DFT) methods were used to quantitatively model the superconductivity in a novel, recently predicted and probably high-pressure formed carbon nanostructure [1]. As the pristine material presents a narrow electronic band gap and an intense peak in the electronic density of states near the Fermi energy, doping was explored to evaluate if this could render the structure superconducting. Computer models reveal that, upon doping with several metallic ions, metallic behaviour is indeed observed; however, most of the doped phases also become dynamically unstable. Doping with Li, Na, Ga, Be atoms were found to yield dynamically stable superconducting structures yet with critical temperatures below 2 K [2].

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High pressure-assisted extraction of phenolic compounds from mango by-products

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Poster Session 1 and Refreshments, July 25, 2023, 16:00–18:00

Introduction: Technologies as high-pressure processing (HPP) and ultrasound-assisted extraction (UAE) in combination with ethanol/water as green solvent were evaluated as sustainable procedures for the recovery of phenolic compounds with antioxidant activity from underutilised fruit by-products (1,2). The combination of HPP and UAE allows the use of room temperature, low volumes of organic solvents, reduction of extraction time, and energetic consumption, with higher yields and high-quality final extracts in comparison with conventional extraction technologies (3).

Materials & Methods: Discarded fully ripped mangos cv Keitt (MK) from Tenerife (Spain) were transported to ICTAN-Madrid, washed, peeled and peel submitted to HPP (200, 400, 600 MPa, 25ºC, 5 min) and then extracted with ethanol/water (50:50) in an US-equipment (500 Watts-20 kHz) at different conditions (Amplitude-A, 60 and 70%; time-t, 10 and 20 min). Total phenolic content (TPC), antioxidant activity (AA) (DPPH•, FRAP) and phenolic profile by HPLC-ESI-QTOF-MS/MS were determined.

Results: Between the MK-peels without HPP (0-MPa), UAE at 70%A-10 min samples presented the highest TPC (13.37 mg GAE/g dw), AA-FRAP (141.50 µmol TEAC/g dw) and AA-DPPH (365 µmol TEAC/g dw). Between HPP-samples, MK-peels treated at 200-MPa showed the maximum significant increase ($p<0.05$) in TPC and DPPH values for all the UAE conditions assayed in comparison with 0-MPA samples. TPC and DPPH increases were: 15% and 41% (70% A-10 min); 29% and 44% (70% A-20 min); 40% and 35% (60% A-10 min) and 44% and 29% (60% A-20 min). The FRAP values increased between 2.5-3.5 times (70% A-20 min) in PK-peels at 400-MPA compared to 0-MPa.The major phenolic compounds found in MK-peel were gallic acid derivatives (gallates and gallotannins).

Conclusion: High pressure processing between 200-400 MPa is an effective pre-treatment for the extraction of phenolic compounds with antioxidant activity from mango by-products as peel in combination with ultrasound-assisted extraction and green solvents.

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High-Pressure/High-Temperature Synthesis and Crystal Structure of two new Zinc Oxotellurate(VI) Compounds

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Poster Session 2 and Refreshments, July 27, 2023, 16:00–17:30

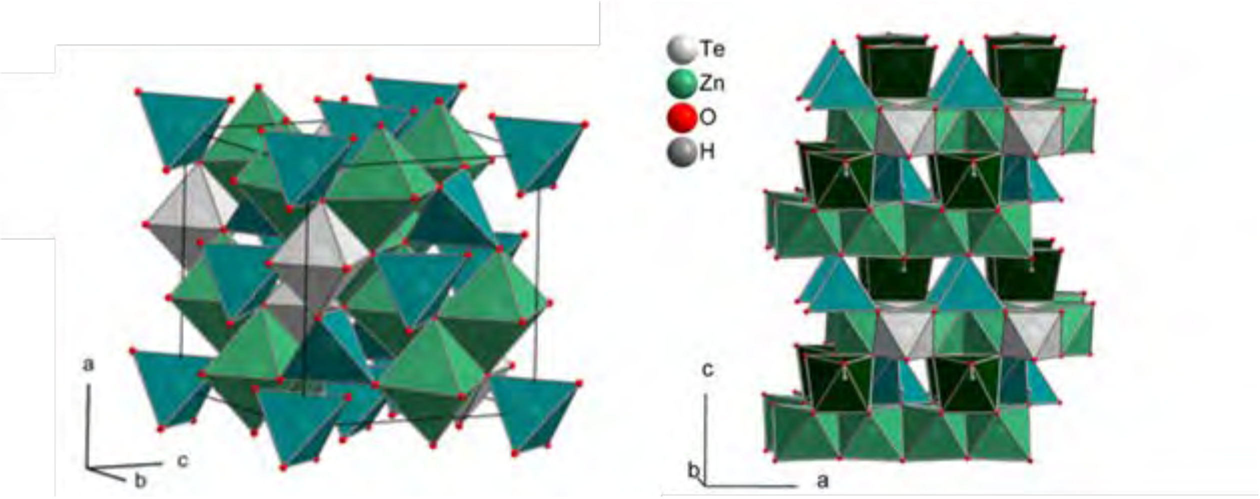
Tellurates have attracted increasing interest in recent years due to their interesting physicochemical features such as nonlinear optical, pyroelectric, piezoelectric or ferroelectric properties. These properties are linked to their corresponding crystal structures, the successful elucidation of which is a crucial prerequisite. To achieve this, we have focused on the synthesis of new tellurate compounds and to optimise the crystal size and crystal quality under multi-anvil high-pressure/high-temperature conditions.

Two new zinc tellurates(VI) have been obtained by experiments at quasi-hydrostatic pressure in a Walker-type module at elevated temperatures.[1] Zn_5TeO_8 was synthesised at 6 GPa and 1073 K and crystallises in the cubic space group $P4_132$ with a unit cell parameter of a = 856.69(1) pm. In this crystal, Te6+ is octahedrally coordinated whereas Zn2+ ions exhibit both octahedral and tetrahedral environments. At higher synthesis pressures of about 7 GPa and temperatures of 1173 K, a second phase in this system has been identified as $Zn_4TeO_6(OH)_2$. The compound crystallises orthorhombically in the space group $Pmn2_1$ with the unit cell parameters a = 595.28(1), b = 538.47(1), c = 949.91(1) pm. In this case, the Te6+ cations are also octahedrally coordinated, but the Zn2+ ions now adopt three different coordination arrangements, ranging from tetrahedral to square pyramidal to octahedral.

Here we present the crystal structures, MAPLE and bond-valence calculations, as well as spectroscopic studies at the two new zinc oxotellurate compounds Zn_5TeO_8 and $Zn_4TeO_6(OH)_2$.

[1] H. Huppertz, Z. Kristallogr. 2004, 219, 330.

Figure: Crystal structure of Zn_5TeO_8 (left) and $Zn_4TeO_6(OH)_2$ (right).



Phosphorus dimerization in GaP under high-pressure

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A new polymorph of GaP with the so called super-Cmcm structure (oS24) similar to that previously described in InSb was recently synthesised at 43 GPa and high temperature, but in GaP the phase forms, surprisingly, single covalent P-P bonds, which is a feature in striking contrast with previous understanding of bonding in the high-pressure polymorphism of III-V compounds. Further investigation on GaP in the range 20-30 GPa has led to the identification of another dimerised stable polymorph with monoclinic symmetry, mS16, which has a new structure type related to super-Cmcm and the well-known Cmcm structure. Thus, two decades after the high-pressure polymorphism of the octect binary semiconductors was considered a substantially settled matter, these findings unravel a marked complexity arising from strong bonding differentiation and finely tuned pressure dependence of the degree of dimerization in the two observed metallic phases of GaP. The discovery of these polymorphs challenges previous views concerning the unfavourable role played by homonuclear bonds in the energetics of the high-pressure phases of the III-V and II-VI binary compounds and further emphasises the role of NaCl-derived layered structures with partial interlayer dimerization as a relevant type of ordering in the high-pressure phases of these compounds. The layer stacking allows for the accommodation of variable degrees of dimerization and thus distinct ordering sequences of such layers, and therefore new structures, are likely to occur in other compounds of the family.

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