MS22 O1

Single crystal diffraction at extreme pressures Michael Hanfland[a], H. Müller[a], K. Syassen[b], [a]*ESRF, Grenoble, France,* [b]*MPI für FKF, Stuttgart, Germany*
E-mail: hanfland@esrf.fr

Keywords: high pressure, single crystal, diffraction

Technical advances have considerably added to the utility of single crystal studies at high pressures. New ways of supporting diamond anvils, like Boehler Almax anvils [1], have significantly increased the volume of accessible reciprocal space. Use of He as pressure transmitting medium extends substantially the practicable pressure rage. Here we will present two examples to illustrate the recent progress.
In the first example single crystals of α-ET_2I_3 [2] were loaded in LeToullec type membrane driven diamond anvil cells, modified for Boehler-Almax anvils with He as pressure transmitting medium. Datasets with 2° rotation images were taken at various pressures to 23 GPa with a Mar image plate scanner on the ID9A beamline of the ESRF. Total rotation range was 60º. The datasets were measured at photon energy of ~30keV and integrated with XDS [3]. The α-phase of ET_2I_3 is stable to 13 GPa. At 13 GPa it undergoes a structural phase transition. The high pressure phase, which remains single crystalline, can be indexed with a triclinic unit cell with twice the volume than that of α-ET_2I_3. The transition is reversible.
The second example deals with the guest host structure observed in Na at pressures above 125 GPa [4].

[1] Boehler R., DeHantsetters K., *High Press. Res.* 2004, 24, 391
[2] Work done in collaboration with H. Müller
[3] W. Kabsch, *J. Appl. Cryst.*, 1993, 26, 795
[4] Work done in collaboration with K. Syassen

MS22 O2

Nanocage materials under high pressure A. San-Miguel, R. Poloni, N. Rey, P. Toulemonde, S. Le Floch, D. Machon and V. Pischedda *LPMCN, University of Lyon and CNRS, Lyon, France.*
E-mail: alfonso.san.miguel@lpmcn.univ-lyon1.fr

Keywords: high pressure, fullerene, clathrate

The study of nanomaterials under extreme conditions constitutes an expanding domain relevant for fundamental science as well as for applications [1]. Nanocage materials are particularly interesting in this context. In fact, their open structures allow for complex host-guest interaction which can be modulated by pressure. On the other side the combination of pressure and temperature can allow for the formation of new assemblages giving rise to novel nanomaterials.
Recent results on two important cases of nanocage intercalated materials will be here discussed: group 14 clathrates and intercalated fullerenes. They are well differentiated systems due to the type of interactions between the nanocages, the size of the cages and the preferred intercalation modes [1]. We have recently reviewed the high pressure properties of group 14 clathrates [2]. The high pressure behavior of silicon clathrates is dominated by the extended stability of the structure due to nanointercalation and by the presence of an homothetic isostructural volume collapse [3] which nature is under debate and is giving rise to an important number of studies and publications. Our discussion will be centered on these aspects.
In the case of intercalated fullerenes, recent results on the construction of the first pressure-temperature phase diagram of alkali intercalated C_{60} fullerenes will be presented.

[1] San-Miguel A., *Chem. Soc. Rev.*, 2006, 35, 876.
[2] San-Miguel A. And Toulemonde P.. *High Pressure Research.* 2005, 25, 159.
[3] A. San-Miguel, A. Merlen, P. Toulemonde, T. Kume, S. Le Floch, A. Aouizerat, S. Pascarelli, G. Aquilanti, O. Mathon, T. Le Bihan, J.P. Itié and S. Yamanaka, *Europhysics Lett*. 2005, 69, 556.

MS22 O3

Pressure-induced phase transitions in hydrides Igor Goncharenko[a], Olga Makarova[b], Michael Hanfland[c], [a]Laboratoire Léon Brillouin CEA-CNRS, 91191 Gif-sur-Yvette, France. [b]Russian Research Center "Kurchatov Institute", 123182 Moscow, Russia. [c]European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France. E-mail: Igor.Goncharenko@cea.fr

Keywords: pressure, diffraction, phase transitions

We report pressure-induced magnetic and structural transitions in hydrides studied by a combination of X-ray and neutron probes in 100 GPa pressure range.
Hydrides of metals exhibit very unusual coupling of structural, magnetic and transport properties. In our previous studies we had reported a pressure induced magnetic collapse in the cubic Laves hydrides formed by rare-earth and transition metals [1,2]. Our recent studies, extended to new type hexagonal hydrides, evidenced new peculiar phenomena. Under high pressure, we observed both "magnetic amorphization" and "magnetic crystallization". The complexity of magnetic phenomena comes from a competition of different interactions and peculiar crystal structure of these compounds.
Whereas the hydrides of rare-earth or transition metals (right-down part of the periodical table) are typically metals, the hydrides formed by elements from the left-upper corner of the periodic table are insulators bonded by ionic or covalent bonds. Under high pressure, these hydrides might form "hydrogen dominant" phases recently suggested by theory [3]. We report new pressure-induced crystal structures and important changes in interatomic bonding in these hydrides.
Possibility to carry out X-ray and neutron measurements on the same sample in the same thermodynamical conditions of pressure and temperature provides new opportunities for high-pressure studies [4], especially for studies of magneto-structural coupling or structural transitions involving light elements. We describe new pressure techniques and discuss further prospects for single-crystal and powder diffraction under very high pressures.

[1] I Goncharenko, P. Cadavez-Peres, , I. Mirebeau, O. Makarova, *Europhys. Lett.,* 2001, 54, 807.

[2] I. Goncharenko, P. Cadavez-Peres, O. Makarova, T. Le Bihan, M. Mezouar, *Phys. Rev. B*, 2003, 68, 214418.
[3] N. Ashcroft, *Phys. Rev. Lett.*, 200492, 187002.
[4] I. Goncharenko, *High Press. Res.,* 2007, 27 183

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Single crystal studies of pure elements at high pressure

Lars F. Lundegaard and Malcolm I. McMahon, *SUPA, School of Physics and Centre for Science at Extreme Conditions, The University of Edinburgh, Edinburgh, UK.* E-mail: lars.lundegaard@ed.ac.uk

Keywords: high-pressure, elements, incommensurate

Although the technique of high-pressure single-crystal diffraction has been used to study pure elements for many years [1], recent developments, such as improved diamond anvil cell design combined with CCD-equipped diffractometers and synchrotron sources, have allowed the structure solution of a number of complex new phases [2,3].
In this talk we will describe the technique of high-pressure single-crystal diffraction, and discuss its advantages and limitations. To Illustrate the power of the technique, we will present recent results on the full modulated structure of the incommensurate composite structure of Rb-IV, the structure of Ba-IVb and the structure of epsilon-oxygen [4].
Finally, we will describe our on-going development of techniques that will enable us to do combined high-pressure high-temperature single-crystal diffraction, and single-crystal diffraction at pressures of 50GPa and above.

[1] D. Schiferl, D. T. Cromer & R. L. Mills, Acta Cryst. (1981), B37, 1329-1332.
[2] M. I. McMahon, R. J. Nelmes & S. Rekhi, Phys. Rev. Lett. (2001), 87, 255502.
[3] R. J. Nelmes, M. I. McMahon, J. S. Loveday & S. Rekhi, Phys. Rev. Lett. (2002), 88, 155503.
[4] L. F. Lundegaard, G. Weck, M. I. McMahon, S. Desgreniers & P. Loubeyre, Nature (2006), 443, 201-204.

MS22 O5

Electric field induced lattice displacements in BiB_3O_6 crystals,

Ullrich Pietsch[a], Oleg Schmidt[a], Semen Gorfman[a], Petra Becker[b], Ladislav Bohaty[b] [a]*Solid State Physics Department of Physics, University of Siegen, Germany.* [b]*Institute of Crystallography, University of Colone, Germany.* E-mail: pietsch@physik.uni-siegen.de

Keywords: Charge density, Piezoelectric crystals, Electric field induced

Monoclinic BiB_3O_6 is a piezoelectric material with exceptional large piezoelectric coefficients [1]. At this material X-ray charge density analysis is highly affected by the huge difference in electronic numbers of constituting elements and by secondary extinction. However, measurements of intensity variation of selected Bragg reflection caused by an external high electric field provide information about the reorganization of atomic positions within the unit cell and subsequently about a change of chemical bonds [2]. For BiB_3O_6 we have measured several Bragg reflections under influence of an external electric field up to 20 kV/cm using synchrotron radiation. The measured change in angular positions of Bragg reflections is caused by the external piezoelectric effect. Measuring the respective peak shifts at three differently oriented crystal plates we have deduced all eight independent coefficients of the piezoelectric tensor of the material [3]. In addition, the intensity is changed due to the internal piezoelectric effect, i.e. the reorganization of atomic arrangement within the unit cell. Preliminary modelling suggested an effect less then 1%. However, tuning the probing wave length close to the absorption edge of Bi atoms we could measure the linear dependence of intensity variation as a function of the applied field at a [100] oriented crystal plate. Due to the small number of measured reflections we used a rough model for data interpretation, a displacement of rigid lattice of BO_3 units against the fixed Bi atoms. Normalized to E=1kV/mm we deduced a displacement vector $\Delta r = -(14,2\ a_1 + 47,5\ a_3)10^{-5}$, given in relative coordinates of the monoclinic system with $a_{1,2,3}$ as unit vectors. Since the external field was applied in direction $E = 0,146\ a_1 + 0,043\ a_3$ the displacement of the negatively charged BO_3 units are directed by an angle of about 75 degree with respect to the plane of positively charged Bi atoms.

[1] Haussühl, S.; Bohaty, L. ; Becker, P.: Piezoelectric and elastic properties of the nonlinear optical material bismuth triborate, BiB3O6. In: Appl. Phys. A 82 (2006),S. 495–502
[2] Gorfman, S. V.; Tsirelson, V. G.; Pucher, A.; Morgenroth, W.; Pietsch, U.: X-ray diffraction by a crystal in a permanent external electric field: electric-field-induced structural response in α-$GaPO_4$. Acta Cryst. A62 (2006) 1-10.
[3] S. Gorfman, O. Schmidt, U. Pietsch, P. Becker, L. Bohaty. X-Ray Diffraction Study of the Piezoelectric Properties of BiB_3O_6 Single Crystals (2007) Z. f. Krist, accepted