MS16 Structure-property relationships in high pressure crystallography

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MS16-O1 The large-volume press at ID06 of the ESRF for structure-properties research at extreme conditions

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Here we highlight, using examples from our in-house and User repertoire, the opportunities for research into the structures, phase transitions, stabilities and properties of materials under extreme conditions. We draw on a range of samples – both powder and single-crystal - and scientific domains to illustrate our talk. We will describe the various modes of operation of the press and the detection system: from 1- and 2-stage compressions with continuous data collections for phase diagram mapping, for estimation of equations of state, for high through-put reconnaissance studies to deformation studies for the collection of the mechanical response of materials under high strain. Highlights of ancillary measurements that augment the information available from diffraction and imaging alone; e.g. ultrasonics will also be commented on.

Keywords: high-pressure, powder diffraction, structures, phase diagrams, materials

MS16-O2 New chemistry of hydrogen-rich systems uncovered with the help of extreme conditions

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The demand for materials with high energy density has shifted the research focus from metal hydrides to much lighter systems based on *p*-elements bound to hydrogen atoms. First seen as merely ionic salts, light complex hydrides based on Al, B and N soon revealed complicated structural chemistry driven by directional interaction between metal cations and complex hydride anions [1]. For example, an unexpected outcome of the directional metal-borohydride interaction are the porous [2, 3] and dense [2, 4, 5] frameworks containing less electropositive Mg and Mn, where the BH_4 units serve as linear linkers.

The dense phases with high volumetric hydrogen density can be obtained by relatively modest hydrostatic pressure. Already at pressures of 1 GPa, achievable in laboratory steel dies, the amorphous $Mg(BH_4)_2$ can be synthesized, as well as the dense form of $Mn(BH_4)_2$. Both phases can be quenched to ambient pressure. The search for the complex order of the BH_4 anions, studied for the first time with neutron powder diffraction under high pressures, will be presented.

The porous hydride phases show remarkable capacity for the loading of guest molecules. In particular, condensation of the molecular hydrogen in the porous γ-$Mg(BH_4)_2$ leads to twice as high hydrogen density in the pores as in the liquid hydrogen, making it the highest local hydrogen density achieved so far in a porous system.

Finally, I would like to draw the attention to the recent developments in the reactive ball milling followed simultaneously by X-ray powder diffraction. This technique allows for *in situ* monitoring the mechano-chemical reactions, including formation of high-pressure phases. Our recent developments (see also the dedicated contribution [6]), involving 3D printing of milling jars, are aiming to improve the data quality for the needs of structure solution.

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Keywords: hydrides, extreme conditions, new materials, porous and dense