

P20.03.17*Acta Cryst.* (2008). A64, C611**Guest disorder, clustering and structure of low/high pressure forms of inert gas clathrate hydrates**Chris A. Tulk¹, Dennis D Klug², Ling Yang¹, Byran Chakoumakos¹, Chris Ratcliffe², John Ripmeester², Igor Moudrakovski², Lars Ehm³, Dave Martin³, John Parise³¹Oak Ridge National Laboratory, Neutron Scattering Sciences Division, Building 8600, Oak Ridge, TN, 37830, USA, ²National Research Council of Canada, ³Stony Brook University, E-mail: tulkca@ornl.gov

Low and high pressure structures of krypton, xenon and argon clathrate hydrates have been studied in situ and in pressure quench recovered forms using high energy (100 keV) synchrotron x-ray scattering (HEXRD) and nuclear magnetic resonance techniques. In addition to standard structural refinements, HEXRD over a wide momentum transfer range has been used to produce Fourier difference maps and pair distribution functions of these structures that indicate the guest electron density. Using relatively simple and higher atomic number inert gas atoms as guests provides enhanced scattering contrast between the guest and the host and allows greater insight into the guest positions and short to intermediate range intercage correlations/disorder of the guest atoms. For krypton hydrate, the low pressure structure II form indicates an enhanced degree of cage-to-cage guest disorder, above that accounted for by ordinary crystalline models. The high pressure hexagonal forms have been produced and successfully quench recovered. The geometry of the large cage guest clusters and intercage guest disorder of this structure are indicated. For the xenon hydrate, we describe the structural refinements for the low pressure structure I form and the in situ high pressure hexagonal form, including descriptions of the guest clustering in the large cages. Structural transformation processes occurring on decompression and recovery of high pressure hexagonal xenon hydrate are also discussed. From the initial low pressure argon hydrate structure I, a filled ice structure is produced above 2.0 GPa, and the structure of this form along with a description of its compressibility is given. ORNL is managed by UT-Battelle, LLC, under contract DE-AC05-00OR22725 for the U.S. Department of Energy.

Keywords: high pressure, disordered materials, clathrates

P20.12.18*Acta Cryst.* (2008). A64, C611**LaueGUI- an open source Matlab tool for online inspection of time resolved Laue diffraction patterns**Marc Messerschmidt^{1,2}, Thomas Tschentscher¹¹DESY-Hamburg, Hasylab, Notkestr. 85, Hamburg, Hamburg, 22607, Germany, ²SLAC-Stanford, 2575 Sand Hill Road, Menlo Park, CA 94025 USA, E-mail: marc.messerschmidt@desy.de

Pink beam Laue diffraction is a powerful tool for determining time-resolved crystallographic structure. The large bandwidth compared to a monochromatic beam allows the simultaneous collection of hundreds of reflections even for moderate size unit cells at fixed orientation. Below 100 X-ray pulses are needed due to the high brightness of the pink beam at a synchrotron. This allows fast data collection and limits sample degradation due to laser excitation. However, there is a large gap between the speed of data collection and the speed of analysis tools. The purpose of LaueGUI is to fill this void by rapidly evaluating time-resolved Laue patterns. The program is based on the use of Precognition (Renz Research), but special care has been taken that the software is self contained in the Matlab environment. LaueGUI is open source written in an object

oriented style. It's controlled through a graphical user interface (GUI) and loads free format ASCII files for many configurations. The code was executed with MAR165 and MAR133 (MAR Research) images but other detector image formats can be supported. Successful analysis was completed in a few seconds per image with data from ESRF[1], KEK-AR[2] and APS(BioCARS)[3]. At ESRF data from TTF-CA single crystals were collected using 50 single pulses of the 16 bunch mode at ID09b (U17, 9mm gap, 18keV, 3% bandwidth) that allowed full structure refinement. Comparable data were collected for organometallic samples at KEK-AR using 3-10 single bunches. Download: userpage.chemie.fu-berlin.de/~mcmesser/LaueGUI references to unpublished data:

[1] M. Messerschmidt, T. Tschentscher, M. Wulff

[2] P. Coppens, M. Gembicky, M. Messerschmidt, M. Pitak, S.-L. Zheng, S.-I. Adachi, S.-Y. Koshihara

[3] P. Coppens, M. Gembicky, M. Messerschmidt, I. Vorontsov

Keywords: time-resolved laue diffraction, crystallographic software development, structural chemistry organic organometallic compou

P20.13.19*Acta Cryst.* (2008). A64, C611**A good bye from SMX @ the SRS**

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For more than ten years SMX has grown to be one of the most powerful techniques and oversubscribed facilities hosted by the Synchrotron Radiation Source (SRS) located at Daresbury, Warrington. In this final review from the SMX stations I will take a look back over the history of both the SMX Stations, 9.8 and 16.2 SMX, highlighting the many successes and the few failings as SMX hands on the batten to another facility and a new generation of experimenter.

Keywords: synchrotron, non ambient, small molecule

P20.03.20*Acta Cryst.* (2008). A64, C611-612**Phase transition studies for powder and amorphous materials under high pressure**Haozhe Liu¹, Luhong Wang¹, Peter Lee², Russell Hemley³, Ho-kwang Mao³¹Harbin Institute of Technology, 2 Yikuang Street, Science Park 2G, Harbin, Heilongjiang, 150080, China, ²Advanced Photon Source, Argonne National Laboratory, Argonne, IL, 60439, USA, ³Geophysical Laboratory, Carnegie Institution of Washington, Washington, DC 20015, USA, E-mail: haozhe@hit.edu.cn

The study of polymorphism and polyamorphism in pressure domain will undoubtedly broaden our horizons and perspectives of the states of matter in general, and may have a significant impact on the existing theories about the structure, formation, and evolution of crystal and amorphous materials. The zinc oxide from NIST standards was studied under high pressure and low temperature conditions. Metallic glasses were selected to test the pressure induced polyamorphism. The procedure of the pressure-induced amorphous state to crystalline state is another subject in this report. The high

energy x-ray diffraction and diamond anvil cell techniques were used to study the structural evolution of powder and amorphous materials under pressure, as well as the time dependence of the crystallization procedure. These will provide new insight on the nature of phase transition, provide new invitation for the electronic theoretical studies for the phase stability, and improve our understanding of the kinetic process of the common pressure induced crystallization.

Keywords: high pressure, phase transition, high energy XRD

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Pressure-induced change of the chemical short-range order in liquid compounds

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To reveal the effect of chemical bonding character on the pressure-induced structural changes, we have investigated the structures of various tetrahedrally bonded materials, such as liquid group 14 elements, III-V, II-VI and I-VII compounds, under high pressure. The results have shown that these liquids show various high-pressure behaviors (local structures and the sharpness of structural changes), depending on the covalent/ionic character [1]. However, these results are based on the average structure, which does not differentiate the chemical component around each atom. To observe partial structures is inevitable to understand the nature of the various structural changes in liquid compounds. For this purpose, we have developed the method to determine the partial structure in liquid compounds at high pressures using an anomalous x-ray scattering method (AXS) and applied it to liquid AgI. The experiment was performed at a JAEA undulator beamline BL22XU in SPring-8. The high pressure and high temperature condition was generated with a multi-anvil high-pressure apparatus, SMAP180. The data was collected by an angular dispersive x-ray diffraction method using a solid state detector. The incident x-ray energy was tuned into the energies below 300 and 20 eV from the absorption edge of two constituents. The obtained partial structural functions are in good agreement with the results of ab-initio MD calculation [2], which supports the validity of the AXS method to investigate the chemical short-range order in the liquid compounds. [1] T. Hattori et al., PRB 68, 224106 (2003), T. Hattori et al., PRB 72, 064205 (2005), T. Hattori et al., PRB 73, 054203 (2006), T. Narushima et al., PRB 76, 104204 (2007), T. Hattori et al., PRB 76, 144206 (2007), [2] F. Shimojo, (private comm.)

Keywords: high pressure, liquid, anomalous x-ray scattering

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A method for analysis of energy-dispersive X-ray diffraction from disordered systems under pressure

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Energy-dispersive x-ray diffraction method is commonly used for x-ray diffraction measurements using a cubic-type multi-anvil-press

because good background elimination and short data acquisition time can be achieved simultaneously. However, diffraction intensity is affected by several energy-dependent factors, such as intensity spectrum of a synchrotron radiation source, sensitivity spectrum of a detector, absorption spectra of the sample and those of the surrounding materials. Because it is difficult to estimate these factors separately, Tsuji et al., proposed a new empirical method for determination of structure factor of liquids and glasses [1]. Furthermore, Funakoshi et al., developed a Monte Carlo simulation code to carry out the procedure [2]. But these analyses sometimes give unreasonable results. We improved the method by implementing a common normalization method in a new code. The obtained structure factors of silica glass and liquid water at ambient conditions agree well with those reported previously.

[1]K. Tsuji, et al., Rev. Sci. Instrum. 60 (1989) 2425.

[2]K. Funakoshi, Ph.D thesis, (1997, Tokyo Institute of Technology).

Keywords: liquid-state diffraction, high-pressure structure determination, energy-dispersive X-ray diffraction

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Pressure- and temperature-induced structural phase transition mechanisms of nitrogen

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A theoretical description is proposed for the pressure and temperature induced structural transitions of nitrogen. Three regions of the phase diagram are distinguished corresponding to different types of transition mechanisms and parent structures. Combined ordering and displacive reconstructive transitions are found in the lowest pressure region, from the parent disordered β structure to the ordered α and γ structures. In a second region, which extends from about 2 to 140 GPa at room temperature, group-subgroup related structures occur, which realize ferroelastic or ferroelectric distortions of the parent δ structure. Space-group symmetries, consistent with the assumed structural mechanisms, are proposed for the δ_{loc} , ζ , κ , ι and θ molecular phases. Above 140 GPa, in the molecular dissociation region, the local structure of the η phase is assumed to realize a link between the molecular ζ and κ phases and the high-temperature cg-N phase.

Keywords: nitrogen, phase transitions in solids, Landau theory

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Thermodynamic exploration of conformational space of 1,2-ethylene glycol

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Ethylene glycol (1,2-Ethanediol, EG) is wide by applied mainly as an anti-freezing additive, but also as a substrate and solvate in many industrial. Owing to the vicinal location of two hydroxyl groups (-OH) in this very simple molecule, EG serves as a model