

resulting large structural models (sets of thousands of atomic coordinates) were subjects of geometrical analyses: distributions of the number of first neighbours, as well as local angular correlations have been calculated. In the cases of molecular liquids investigated (like the case of liquid O₂), correlations between orientations of neighbouring molecules have been characterized.

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Keywords: high-pressure X-ray diffraction, diamond anvil cells, computer modelling liquids

MS68.28.4

Acta Cryst. (2005). A61, C88

X-ray Diffraction Study in Liquid Cs up to 9.8 GPa

Sara Falconi^a, Lars Fahl Lundegaard^a, Clivia Hejny^a, Malcolm McMahon^a, ^a*School of Physics and Centre for Science at Extreme Conditions, The University of Edinburgh, Mayfield Road, Edinburgh, EH9 3JZ, United Kingdom.* E-mail: sfalconi@ph.ed.ac.uk

An x-ray diffraction study of liquid Cs at high pressure and temperature has been conducted in order to characterize the structural changes associated with the complex melting curve and phase transitions observed in the solid phases [1, 2, 3, 4]. At 3.9 GPa a discontinuity in the density of the liquid accompanied by a decrease in the coordination number from about 12 to 8 marks a change between simple and non-simple liquid regime [5].

The specific volume of liquid Cs, combined with structural analysis of the diffraction data, strongly suggest the existence of an electronic hybridization above 3.9 GPa similar to that reported on compression in the crystalline phase.

Further, *ab-initio* calculations with cluster of 264 atoms confirm the structural results obtained experimentally and reinforce the hypothesis of the electronic hybridization driven by compression of the solid and of the liquid.

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Keywords: caesium, high pressure, liquids

MS68.28.5

Acta Cryst. (2005). A61, C88

CdSb under Pressure: Compound Decomposition, New Phase Formation and Amorphization

Valentina F. Degtyareva^a, Olga Degtyareva^b, Russell J. Hemley^b, ^a*Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka, Russia.* ^b*Geophysical Laboratory, Carnegie Institution of Washington, USA.* E-mail: degtyar@issp.ac.ru

Application of high pressure offers wide possibilities to produce new materials in crystalline and amorphous states. The Cd-Sb alloy system has at ambient pressure one intermediate compound of equiatomic composition, CdSb, orthorhombic, space group *Pbca*, with 16 atoms in the unit cell. On pressure increase to 7.3 GPa, we observed a transition from CdSb-*oP16* phase to a new state that is interpreted as a two-phase mixture of a simple hexagonal Sb-rich phase and a hexagonal close packed phase of (almost pure) Cd. At 8.4 GPa, lattice parameters are for the *sh* phase (space group *P6/mmm*) $a = 3.066(1)$ Å and $c = 2.860(1)$ Å, and for Cd-*hcp* phase (space group *P6₃/mmc*) $a = 2.93(1)$ Å and $c = 5.165(1)$ Å, close to those reported for pure Cd at this pressure. On pressure decrease, the two phase mixture state is observed down to 1 GPa and below 1 GPa, an amorphous phase is observed. The halos of the amorphous phase of CdSb sample correspond to $Q_1 = 2.004$ Å⁻¹ and $Q_2 = 2.953$ Å⁻¹. The full width at half maximum of the halos corresponds to the correlation length 12-15 Å. The amorphous phases formed in binary alloys Zn-Sb, Cd-Sb and Al-Ge after pressure action are close to tetrahedral nets and correspond to nearly 4 el./atom composition [1,2].

Partial support from RFBR under grant 04-02-17343 is acknowledged.

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Keywords: high-pressure phases, decomposition, amorphization

MS69 ELECTRON CRYSTALLOGRAPHY ON ORGANIC CRYSTALS AND BIOMOLECULES

Chairpersons: Hans Hebert, Ute Kolb

MS69.28.1

Acta Cryst. (2005). A61, C88

Electron Diffraction from a Beam of Laser-aligned Proteins: Progress Report

Uwe Weierstall, Dmitri Starodub, John C.H. Spence, Bruce Doak, *Department of Physics and Astronomy, Arizona State University, Tempe AZ 85287-1504, USA.* E-mail: weier@asu.edu

The use of diffraction patterns from aligned molecular beams has been proposed as an approach to the structure determination of proteins that are difficult to crystallize [1]. Polarized laser light is used to align the molecules in the beam. By cooling a beam of hydrated proteins to low temperature, sufficient alignment for diffraction purposes might be possible. Therefore an arrangement has been suggested [2] consisting of continuous, orthogonal, intersecting electron (or X-ray), molecular and laser beams. Limited coherence of the electron beam ensures that no interference occurs between the wavefields scattered by different molecules within the electron beam. The diffraction pattern is a sum of the intensities of the identical patterns from the many hydrated molecules within the beam at any one time. The two-dimensional electron diffraction pattern accumulates continuously at the detector for a fixed laser and molecular orientation before being read out. Repeating this process for many orientations would allow tomographic reconstruction of the molecule. An electron diffraction camera with a water droplet source, a polarized laser beam and a LaB₆ electron gun is currently under development. Initially we use micron size water droplets and dope them with large particles like TMV, since they are easier to align. Preliminary results with this instrument will be presented.

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Keywords: electron diffraction, protein structure determination, image reconstruction

MS69.28.2

Acta Cryst. (2005). A61, C88-C89

Methods Development and Software Engineering for 2D Electron Crystallography

Ansgar Philippsen^a, Andreas Schenk, Giani Signorell, Wanda Kukulski, Andreas Engel, ^a*Maurice Müller Institute for Structural Biology, Biozentrum, University of Basel, Switzerland.* E-mail: ansgar.philippsen@unibas.ch

Electron crystallography of 2D crystals (2D-EX) of membrane proteins has allowed atomic models of these proteins to be built (1,2). Several advantages of this method over 3D x-ray crystallography can be identified, such as preserving the protein in a more native state or imaging of the crystal with direct amplitude and phase retrieval. Nevertheless, this method has not yet matured to the point of routine use. We pursue the goal to establish 2D-EX membrane protein crystals as a complementary method (next to x-ray crystallography and structural NMR) for resolving atomic resolution structures of biological macromolecules. To this end, we are in the process of implementing and refining the methods utilized by the pioneers of the field (1,2), as well as following novel methodological and algorithmic approaches. In parallel, we are developing an image processing library and toolkit (3), tailored to the specific requirements of these methods.

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Struct. Biol., 2003, **144**, 4.

Keywords: membrane proteins, electron crystallography, methods development

MS69.28.3

Acta Cryst. (2005). A61, C89

Atomic Model of Microsomal Glutathione Transferase 1 from Electron Crystallography

Peter J Holm^{a,b}, Priyaranjan Bhakat^b, Caroline Jegerschöld^b, Nobuhiko Gyobu^c, Kaoru Mitsuoka^c, Yoshinori Fujiyoshi^c, Ralf Morgenstern^a and Hans Hebert^{a,b}, ^aKarolinska Institutet, Sweden. ^bLund University, Sweden. ^cKyoto University, Japan. E-mail: peter.holm@mbfys.lu.se

The integral membrane protein microsomal glutathione transferase 1 (MGST1) possesses glutathione and peroxidase activity thus protecting the organism from toxic substances. We have determined the atomic model of MGST1 at 3.5Å resolution by electron crystallography of 2-dimensional crystals from two different two-sided plane groups making it the first membrane enzyme solved to atomic resolution by this technique. The MGST1 homotrimer is constructed by 12 trans-membrane helices forming three all alpha-up-down 4-helix bundles with a fold strikingly similar to the cytochrome c oxidase subunit I suggesting divergent evolution from a common structural ancestor. The MGST1 model reveals inter-subunit interaction and strengthens previous suggestions of global conformational changes upon glutathione (GSH) binding. Furthermore a possible location of the putative hydrophobic binding site is suggested.

Keywords: microsomal glutathione transferase 1, membrane protein structure, electron crystallography

MS69.28.4

Acta Cryst. (2005). A61, C89

Structural Features of Cyclodextrin Inclusion Complexes

Akiyoshi Kawaguchi, Junka Kawasaki, *The faculty of science and engineering, Ritsumeikan University, 1-1-1 Nojihigashi, Kusatsu, Shiga 525-8577, Japan.* E-mail: akiyoshi@se.ritsumei.ac.jp

Inclusion complexes of α -, β - and γ -cyclodextrins with poly(ethylene glycol)(PEG) and other polymers were prepared using various methods reported so far. The inclusion complexes exhibited the following crystalline features.

1. The inclusion complex of α -cyclodextrin/poly(ethylene glycol) gave a very spotty hexagonal electron diffraction pattern, which gave the hexagonal unit cell. In the case of β -cyclodextrin/ poly(propylene glycol), a spotty hexagonal electron diffraction pattern was also observed. The spotty appearance of these electron diffraction pattern is caused by the following host/guest arrangement: The host cyclodextrin columns are arranged in ordered way, even though guest molecules randomly oriented in the caves of dextrin hosts.

2. The inclusion complex of γ -cyclodextrin with poly(ethylene adipate) gave a "superlattice" comprising 8x8 cyclodextrin units, and additionally its electron diffraction pattern showed the characteristic streaky diffuse scattering due to the attacking fault of the cyclodextrin units.

3. The inclusion complexes contained water molecules in them. The crystal structure was largely disordered, when water molecules were removed by heat treatment. As the original structure was recovered by exposing water vapor, the structural order/disorder transition occurs reversibly.

Keywords: cyclodextrin, superlattice, electron diffraction

MS70 NEW SAS FRONTIERS

Chairpersons: Gernot Kosterz, Theyencheri Narayanan

MS70.28.1

Acta Cryst. (2005). A61, C89

In-situ X-ray Scattering Studies of Nanomaterial Growth Dynamics in Aerosols

Gregory Beaucage^a, H. K. Kammler^b, R. Jossen^b, S. E. Pratsinis^b, T. Narayanan^c, J. Ilavsky^d, P. Jemian^d, D. Londono^c, ^aDepartment of

Chemical and Materials Engineering, University of Cincinnati, Cincinnati, Ohio 45221-0012 USA. ^bETHZ Zurich Switzerland. ^cESRF Grenoble France. ^dUNICAT, APS Argonne IL USA. ^eDupont Corporation, Wilmington DE USA. E-mail: beaucage@uc.edu

Nanomaterials display unique properties intermediate between those of the molecular and macroscopic regimes. The interest in application of nanomaterials has driven a desire to understand the fundamental mechanisms and processes involved in nanomaterial formation. Generally, nanomaterials are formed under non-equilibrium conditions with deep supersaturation and are commonly formed by extremely rapid growth processes that lead to kinetically dominated structural features. Aerosols offer transient and dramatic changes in temperature, concentration and stoichiometry that can be put to use to produce highly non-equilibrium conditions for nanomaterial formation. Our understanding of nanomaterial formation under these conditions can be studied in situ using synchrotron based techniques. This presentation will highlight some of the most important discoveries made during the past 2 years at ESRF and APS (USA) on flames and environmental aerosols. The work was supported by the Swiss National Science Foundation, the US National Science Foundation and Dupont Corporation.

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Keywords: nanomaterials, nucleation-and-growth, aggregation

MS70.28.2

Acta Cryst. (2005). A61, C89

Dynamics of Amphiphilic Systems Probed by Highly Time-resolved SAS Experiments

Michael Gradzielski^a, Isabelle Grillo^b, Theyencheri Narayanan^c, ^aStranski-Laboratorium, Technische Universität Berlin, Germany. ^bILL, Grenoble, France. ^cESRF, Grenoble, France. E-mail: michael.gradzielski@tu-berlin.de

Amphiphilic molecules form a large variety of self-aggregating structures that are highly dynamic with time scales ranging from μ s to weeks. Often morphological changes can be triggered by mixing with other surfactants, additives, or solubilisates. In our experiments rapid mixing was studied by coupling the stopped-flow technique to high-flux SANS/SAXS instruments which allows to obtain detailed structural information with a time-resolution of 5-50 ms. By this method a large variety of different structural transitions were investigated, e. g. the formation of unilamellar vesicles by admixing oppositely charged surfactant or a cosurfactant. For both cases slow formation of monodisperse unilamellar vesicles is observed that takes place in a way purely governed by diffusion. Both, kinetics and the final structure depend strongly on the electrostatic conditions of the system. In other experiments the disintegration of micelles when mixing with a bad solvent was followed, which passes through a minimum aggregation stage before smaller micellar structures are reformed. This applies also to much larger block copolymer micelles of the PIB-PAA type. Their response to changes of ionic strength and also their complexation with oppositely charged polyelectrolytes was studied. For all cases the details of the transformation can be studied and in particular it is possible to identify intermediate structures, a point which is very important for a systematic control of the dynamics of self-aggregating systems.

Keywords: colloids, amphiphilic molecules, dynamics

MS70.28.3

Acta Cryst. (2005). A61, C89-C90

Cytochrome C and α TS Folding Probed by Submillisecond Continuous-Flow SAXS

Elena Kondrashkina^a, O. Bilsel^b, C. Kayatekin^b, T. Irving^a, C.R. Matthews^b, ^aBCPS Department, Illinois Institute of Technology, Chicago, USA. ^bDepartment of Biochemistry and Molecular