

**MS39.26.3***Acta Cryst.* (2005). A61, C54**Protein Powder Diffraction: why Bother?**Christopher Gilmore, Wei Dong, *Department of Chemistry, University of Glasgow, Glasgow, UK.* E-mail: chris@chem.gla.ac.uk

This discussion of powder protein powder diffraction looks at what is currently achieved with small molecule powder data, and low resolution single crystal protein crystallography and asks what techniques from these areas can be translated into the world of protein powders:

1. Qualitative PXRD: Pattern matching - can we classify and match patterns using the full pattern profile and not just the peaks as in the SNAP-1D [1] and PolySNAP [2] computer software, and can this information be used as an aid to crystallization?

2. Quantitative PXRD: Can we identify components in powders in a quantitative mode using full powder profiles as used in the SNAP-1D/PolySNAP software?

3. Unit Cells with protein PXRD: Can we index poor quality patterns? Can using the full profile help? What about brute force methods using grid computing techniques?

4. Single crystal low resolution protein diffraction can give the molecular envelope; can this be achieved with powder data?

All these issues will be discussed with examples where possible.

[1] Gilmore C.J., Barr G., Paisley J., *J. Appl. Cryst.*, 2004, **37**, 231-242. [2] Barr G., Dong W., Gilmore C.J., *J. Appl. Cryst.*, 2004, **37**, 243-252. [3] Von Dreele R.B., *Acta Cryst.*, 2005, **D61**, 22-32. [4] Gilmore C.J., Wright J., Fitch A., *Transactions of the Amer. Cryst. Assocn.*, 2002, **37**, 113-123.

**Keywords:** protein powder diffraction, pattern matching, envelopes

**MS39.26.4***Acta Cryst.* (2005). A61, C54**Development of Powder Diffraction Methods for Macromolecular Crystallography**

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Modern developments of the powder diffraction technique have allowed the investigation of systems with large unit cells like proteins [1]. Powder diffraction measurements can give a range of complementary information beyond that which can be obtained from a single crystal. For example, the peak shapes depend on the microstructure of the material, accurate unit cell parameters can easily be determined, and the sample generally survives under more varied or extreme conditions. In the present work, we aim in establishing the full potential of powder diffraction technique in the research of macromolecular systems. Specific examples that will be presented refer to: **(a)** structural modifications of the hen and turkey egg-white lysozymes (HEWL & TEWL) with crystallisation conditions and temperature [2-3]. **(b)** in-situ observation of crystal growth of HEWL **(c)** the development of a successful cryoprotection protocol for powder diffraction, with an almost complete suppression of radiation damage in pancreatic porcine elastase & **(d)** heavy atom derivatives of HEWL and elastase. A key point of the current work is to carefully assess various instrumental configurations and experimental strategies for the recording of protein powder data, either with high-resolution scanning instruments or with area detectors.

[1] Von Dreele R. B. *Acta Cryst.*, 2005, **D61**, 22-32. [2] a) Margiolaki I., et al., *Acta Cryst.*, 2005, **D61**, in press; b) see also: *ESRF Scientific Highlights*, 2004, 24. [3] Basso S. et al., in preparation.

**Keywords:** proteins, powder diffraction, synchrotron radiation

**MS39.26.5***Acta Cryst.* (2005). A61, C54**Protein Measurements on a Laboratory Powder Diffractometer**Stjepan Prugovečki<sup>a</sup>, Detlef Beckers<sup>a</sup>, Thomas Degen<sup>a</sup>, Biserka

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Han egg white lysozyme has been dissolved into 0.1 M sodium acetate-acetic buffer at pH 4.8 to a final concentration of 60mg/ml. This has been mixed with a second solution of 8% sodium chloride in the same 0.1 M puffer and kept at 22°C. Tetragonal crystals of various sizes smaller than 150 µm were obtained after 46 hours. These crystals were removed from the mother-liquor using a pipette, and placed into a 0,5mm glass capillary. The crystals were manually compacted and the remaining mother-liquor has been removed.

The sample was measured on an X'Pert PRO Multi-Purpose Diffractometer (MPD), configured in transmission geometry with a focusing mirror on the incident beam side and an X'Celerator detector on the diffracted beam side. The sample was measured at room temperature with a scan range of 1-45° (2θ), a step size of 0.004°.

The raw data were indexed by the DICVOL program, integrated into the HighScore Plus software suite, giving a tetragonal cell with parameters: a= 79.09Å, c=37.94Å. Le Bail and Pawley fit, as well as structural refinement showed good agreement and results will be discussed.

**Keywords:** proteins, powder X-ray diffraction, X-ray optics

**MS40 COMPUTATIONAL CRYSTALLOGRAPHY APPLIED TO EXTREME CONDITIONS***Chairpersons:* Giulia Galli, Alessandro Pavese**MS40.26.1***Acta Cryst.* (2005). A61, C54**Structural Paths for the High-Pressure Phase Transitions of AgI**Michele Catti, *Dipartimento di Scienza dei Materiali, Università di Milano Bicocca, Italy.* E-mail: catti@mater.unimib.it

First-principles calculations with Density-Functional-Theory (DFT) Hamiltonian and localized basis set (CRYSTAL code [1]) were performed on AgI, showing that it transforms from the cubic zinc blende to the tetragonal anti-litharge structure at 1.2 GPa, and then to cubic rocksalt at 1.6 GPa, in agreement with experiment [2]. For both reconstructive phase transitions, a monoclinic *Pm* pathway was considered and tested by computing the enthalpy profile (with full structural optimization) vs. the order parameter, according to a previously presented method [3]. On the basis of the activation enthalpies obtained, a bifurcated three-step kinetic mechanism is proposed. One step relates the anti-litharge structure to a metastable orthorhombic *Bmm2* phase which appears along the transformation path. Then two alternative steps follow, transforming the intermediate phase into either zinc blende or rocksalt. The enthalpy curve along the *Pm* pathway shows two bottle-neck states bracketing the metastable phase, with a predicted maximum activation enthalpy of 0.088 eV. The mechanism is characterized by changes of the Ag coordination number from 4 (zinc blende and anti-litharge) to 5 (*Bmm2* phase) to 6 (rocksalt), which account for the dependence of the unit-cell volume on the order parameter.

[1] Saunders V.R., et al., *CRYSTAL03: User's manual*, 2003, University of Torino, Italy, and CLRC Daresbury Laboratory, UK. [2] Keen D.A., Hull S., *J. Phys.: Condens. Matter*, 1993, **5**, 23. [3] Catti M., *Phys. Rev. Lett.*, 2001, **87**, 035504.

**Keywords:** high-pressure phase transformations, ab initio calculations, phase transition kinetics

**MS40.26.2***Acta Cryst.* (2005). A61, C54-C55**The Fifth Element in the Periodic Table, Boron: do we know the Ground State Structure?**Tadashi Ogitsu, Giulia Galli, Francois Gygi, *Lawrence Livermore National Laboratory, University of California, USA.* E-mail: ogitsu@llnl.gov

Boron exhibits the most complex structure of all elemental solids, with more than 300 atoms per unit cell arranged in interconnecting

icosahedra. The best estimate on the number of atoms in the unit-cell is not even an integer number, 320.1, originating from the introduction of partial occupancy of atomic sites in the X-ray structural analysis [1].

This work is the first attempt, using *ab initio* molecular dynamics, to study the stable configuration of the partially occupied sites (POS) in  $\beta$ -boron and to investigate POS impact on the electronic structure. We have found that the correlated POS configurations not only lower the total energy of the solid, but also widen the electronic band gap, giving consistent results with experiments.

The high pressure phases of boron[2,3] have also been studied with *ab initio* simulated annealing methods. We found that at around 120GPa,  $\beta$ -rhombohedral boron undergoes amorphization and that its electronic conductivity rises, due to delocalization of the electronics states near the Fermi level, consistent with experimental observations[2,3].

This work was performed under the auspices of the U. S. Dept. Energy at the University of California/LLNL under contract no. W-7450-Eng-48.

[1] Slack G. A., et al., *J. Solid State Chem.*, 1988, **76**, 52. [2] Eremets M. I., et al., *Science*, 2001, **293**, 272. [3] Sanz D. N., et al., *Phys. Rev. Lett.* 2002, **89**, 245501.

**Keywords:** *ab initio* structural determination, high pressure structure, electronic structure

#### MS40.26.3

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#### Oxides Under Pressure: from Densified Silica to the Rheology of the Earth's Mantle

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The paper will describe recent advances in the atomistic simulation of oxides at extreme conditions of pressure. The simulations are carried out using interatomic force fields optimized by best fit on first-principles (density-functional theory) calculations. The paper will focus on two applications of the method: (a) the mechanisms of permanent densification in silica glass, and (b) the properties of dislocations in MgO, the second most abundant mineral in the Earth's lower mantle.

**Keywords:** simulation, DFT, high pressure

#### MS40.26.4

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#### Novel High-pressure Phases: Theory and Experiment

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Searching for new materials and new crystal structures at high pressures and temperatures is important for fundamental physics, for material sciences, and for understanding the structure and properties of planetary interiors. State-of-the-art computer simulations can fruitfully complement or even guide experimental efforts in this direction. Here, we present recent joint theoretical/experimental discoveries of new geophysically important phases of MgSiO<sub>3</sub> [1-3] and Al<sub>2</sub>O<sub>3</sub> [4] with implications for the structure, dynamics, electrical conductivity, rheology and seismic signatures of the Earth's lowermost mantle.

[1] Oganov A.R., Ono S., *Nature*, 2004, **430**, 445. [2] Murakami M., et al., *Science*, 2004, **304**, 855. [3] Oganov A.R., Martonak R., Laio A., Raiteri P., Parrinello M., 2005, *in preparation*. [4] Oganov A.R., Ono S., *Proc. Natl. Acad. Sci.*, 2005, *submitted*.

**Keywords:** high pressure, *ab initio*, Earth's mantle

#### MS40.26.5

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#### MgSiO<sub>3</sub> Post-perovskite at D" Conditions

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The thermoelastic properties of the newly found post-perovskite polymorph of MgSiO<sub>3</sub>, more stable than the Pbnm-perovskite phase at conditions close to those expected in Earth's D" region, has been investigated by first-principles and contrasted with those of the perovskite phase. We predict the major seismic trends such as velocity discontinuities, ratios of velocities and density anomalies, and anisotropy in aggregates with preferred orientation that should occur in the presence of this phase change. Consequences of this model mineralogy for the D" region will be discussed.

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**Keywords:** phase transition, mantle mineralogy, thermoelasticity

#### MS41 COMPUTATIONAL SOLUTIONS FOR HIGH-THROUGHPUT CRYSTALLOGRAPHY

*Chairpersons:* Duncan E. McRee, James Holton

#### MS41.26.1

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#### Crank - New Methods in Automated Structure Solution

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We present Crank[1], a suite to help a user perform automated macromolecular structure solution. In this aim, it uses novel programs, including CRUNCH2 for substructure determination and BP3 for substructure refinement and phasing. In addition, Crank uses other commonly used crystallographic programs including SOLOMON, DM and various programs within the CCP4 suite. Crank uses the CCP4i package for its user interface, this allows for tight integration into the CCP4 suite and presents the user with a familiar interface. Crank uses the XML eXtensible Markup Language to store, manipulate and compare data, this XML can subsequently be used to assist in data deposition. We have tested Crank on a large number of datasets, including datasets from the Joint Center for Structural Genomics, our results show that Crank often outperforms existing automated substructure solution packages, and can lead to solutions where existing methods fail. For more information, please visit the Crank web site: <http://www.bfsc.leidenuniv.nl/software/crank>.

[1] Ness S. R., de Graaff R.A.G., Abrahams J. P., Pannu N.S., *Structure*, **12**, 1753-1761.

**Keywords:** automated macromolecular structure solution, BP3, crunch2

#### MS41.26.2

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#### Parallel Data Processing for High Throughput X-ray Structure Determination

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The Structure Determination Core (SDC) of the Joint Center for the Structural Genomics (JCSG) has implemented a prototype system, Xsolve, which automates all of the processing steps needed to create an initial set of molecular coordinates from a dataset of diffraction images. The goal of Xsolve is to provide standardized, high quality data processing and automate the numerous time-consuming steps in the structure determination process. The current prototype produces a model that is over 95% complete in more than 80 % of the MAD cases