

**P02.16.55***Acta Cryst.* (2008). A64, C217**Structure solution of Ag(py<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) in the presence of impurity phases using robust refinement**Kevin H Stone<sup>1</sup>, Saul Lapidus<sup>1</sup>, Peter W Stephens<sup>1</sup>, Jamie Manson<sup>2</sup><sup>1</sup>Stony Brook University, Physics and Astronomy, State University of New York at Stony Brook, Stony Brook, NY, 11794-3800, USA, <sup>2</sup>Department of Chemistry and Biochemistry, Eastern Washington University, Cheney, WA, 99004-2440, USA, E-mail: khstone@ic.sunysb.edu

High resolution powder diffraction was used to characterize a sample of Ag(py<sub>2</sub>S<sub>2</sub>O<sub>8</sub>). Measurements were taken at beamline X16c at the National Synchrotron Light Source at Brookhaven National Laboratory. The sample was found to contain multiple phases. Subsequent measurements, several months later, showed that a number of diffraction peaks had decreased dramatically in intensity. This is indicative that one phase had partially transformed, presumably into another, already present, phase. The disappearing peaks were indexed as c-centered monoclinic, with lattice parameters  $a = 15.969 \text{ \AA}$ ,  $b = 7.133 \text{ \AA}$ ,  $c = 14.586 \text{ \AA}$ ,  $\beta = 124.971^\circ$ , and a candidate structure found. Due to the presence of multiple phases in the sample, however, ordinary Rietveld refinement of this structure was impossible. A robust refinement<sup>1</sup> procedure was implemented to refine the structure of the disappearing phase in the presence of other, unknown, phases. This was done using the software TOPAS Academic by continuous readjustment of the weights used in the figure of merit calculations, effectively changing  $\chi^2$  into its robust equivalent. The refined structure contains 4-coordinated silver, bonded in a square planar configuration to the nitrogen atoms of the pyrazine rings. The peroxodisulfate ions are located between the sheets of Ag(py<sub>2</sub>S<sub>2</sub>O<sub>8</sub>). The robustly refined structure will be presented, and compared to previous knowledge of this material.

<sup>1</sup> David, W. I. F., *J. Appl. Cryst.* (2001). 34, 691-698

Keywords: powder diffraction, robust refinement, structure solution

**P02.17.56***Acta Cryst.* (2008). A64, C217**Solving oxide structures by precession electron diffraction**

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Oxides are a large family of materials which cover different physical properties potentially interesting for applications including high Tc superconductors, catalysts, multiferroics, solid electrolytes for fuel cells etc. New oxide phases are often obtained as powders either due to the synthesis conditions (HP, HT) or voluntarily in order to exploit the special properties stemming from nanometer sized particles. As a consequence the structure of these materials often is not accessible by X-ray diffraction. Precession electron diffraction (PED) can then be a powerful tool for the structure solution of unknown phases. In this work we present the structures of several oxide phases solved ab initio by PED. We will discuss the influence of different experimental parameters and of the data treatment on the structure solution obtained. Experimentally, a large precession angle is more efficient for reducing the multiple scattering, which is the main advantage in using PED as compared to standard SAED. However, it implies also electron beams that are far off the optical axis of the microscope. These beams are subjected to the lens aberrations more strongly than near axis beams, which reduces the quality of the data. In the data

treatment we investigated the choice of the resolution in reciprocal space. While maximum resolution is desirable, the reflections at the highest resolution are more strongly subjected to the lens aberrations and they may suffer from overlap with the first order Laue zone. The number and the kind of zone axes used is a question of efficiency and/or experimental accessibility. When the sample consists of platelet shaped particles with the same crystallographic axis perpendicular to the platelets, the accessible information along this axis is limited.

Keywords: electron crystallography, TEM, precession electron diffraction

**P02.17.57***Acta Cryst.* (2008). A64, C217**Applying parallel computing for a faster and better structure solution**Zheng-Qing Fu<sup>1,2</sup>, John Chrzas<sup>1,2</sup>, James Fait<sup>1,2</sup>, Zhongmin Jin<sup>1,2</sup>, Andrew Howard<sup>1,3</sup>, John Gonczy<sup>1,2</sup>, John P Rose<sup>1,2</sup>, Bi-Cheng Wang<sup>1,2</sup><sup>1</sup>SER-CAT, APS, Argonne National Laboratory, Building 436, Argonne, IL, 60585, USA, <sup>2</sup>Dept. of Biochemistry and Mol. Biology, Univ. of Georgia, Athens, GA, USA, <sup>3</sup>Biological, Chemical, and Physical Sciences Dept., Illinois Institute of Technology, Chicago, IL, USA, E-mail: fuzq@anl.gov

Given data acquired, there are currently varieties of methods, computing algorithms and programs available in every step of the X-ray crystallographic structure-solving process. Some of them may outperform others in certain cases, and vice versa. In addition, a crystallographic software normally has a lot of controlling parameters for users to twist to get the reasonable result. From reduced data, the current common practice is to try a group of personally favorite programs with various hand-picked parameter combinations until an initial model and traceable electron density map is obtained. For easy cases with high-quality data, it could be straight forward. For an average-quality data set, it would need both experiences and efforts to reach a reasonably good initial model and density map. In difficult cases or when the data quality is marginal, this process may become so tedious that patience could be lost even before finding any solution. Parallel computing, especially powered by a Linux cluster that has become cheaper and ever more affordable, could help turn such a tedious trial-and-error process into a few mouse clicks and quickly produce the best result for a given data. Here we present the design and development of a parallel workflow engine, built inside SGXPRO that can automatically manage communication among different steps, search algorithm & parameter space to find the best solution for a solvable data.

Keywords: parallel algorithms, crystallographic methodology computing, macromolecular structure determination

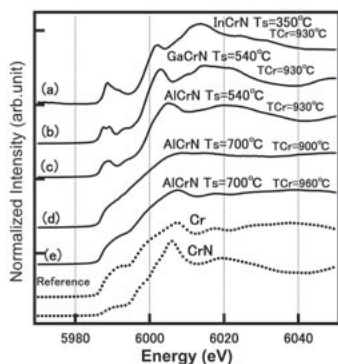
**P02.07.58***Acta Cryst.* (2008). A64, C217-218**Structure properties of AlCrN, GaCrN, and InCrN**

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III-nitride based diluted magnetic semiconductors (DMSs) are attractive materials for applications in spin-dependent electronic devices operating at above room temperature. Here, we will discuss

about growth mechanisms and structural properties of III(Al, Ga, and In)-Cr-Nitride. Those layers were grown on Al<sub>2</sub>O<sub>3</sub> (0001) with GaN (0001) templates by radio frequency plasma assisted molecular beam epitaxy. Structural properties were studied by XANES and XAFS analysis. Fig.1 shows XANES spectra. Cr foil and CrN spectra also are given. In Fig.1, the comparison of the spectra (d) and (e) of AlCrN grown at 973K with that of Cr foil indicates that the Cr clusters with a nano-size, which could not be detected in XRD, are formed in AlCrN grown at 973K. In the other hand, we have reported Cr atoms substitute Ga-site in GaCrN grown at 813K (b). Comparing the spectra (b) to (c), Cr atoms substitute Al-site in AlCrN grown at 813K. In a similar way, Cr atoms substitute In-site in InCrN grown at 813K (a). Low temperature MBE enables the growth of AlCrN, GaCrN and InCrN without CrN segregation or Cr cluster. The structural properties and electric states will be discussed in the conference in detail.



Keywords: diluted magnetic semiconductor, XAFS, III-Cr-N

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### Efforts to improve the phase convergence of the shake-and-bake (SnB) algorithm towards solutions

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“Shake and Bake” procedures attempt to determine crystal structures from random atom starting models by phase refinement algorithms that operate in both real and reciprocal space. The reciprocal space module uses a parameter shift procedure to minimize R<sub>min</sub> for the individual phases for each real/reciprocal space refinement cycle. The majority of the reciprocal space refinement cycles, however, do not actually move the phases towards the solution, but rather perturb the phases so they might escape from local false minima until by chance a downhill pathway to the solution is found. Recent work has surprisingly shown that it is possible to analyze these intermediate non-solution stages of the refinement to identify subgroups of phases that have a significantly lower mean phase error than the remaining reflections in the direct methods trial sets. We are currently examining various methods to exploit this information and accelerate the rate convergence of the SnB process towards solutions. This will be exceedingly important for the more intransigent structure determinations in which all trials constantly languish in the non-solution R<sub>min</sub> optimization state. Research support from the Human Frontier Science Program (HFSP) is most gratefully acknowledged.

Keywords: direct methods, shake-and-bake algorithm, phase refinement

## P03.01.01

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### NRCVAX revisited: Reusing existing software

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This project came about after trying to use a commercial package in a service environment. The software provided, although functional, proved cumbersome especially once one tried to ensure that the resulting structures were easily publishable using CifCheck and could be formatted as readable reports for the users. The intent is to produce a flexible program system that provides error checking throughout the structure solution and refinement process thereby reducing the problems that have to be handled during the report production process. The NRCVAX software package(1) was designed to run interactively on small computers. Most other programs at the time were batch oriented and designed for mainframes, although in later years many of them have been wrapped in interactive user interfaces. One of the more obvious differences was that NRCVAX was a set of discrete programs that each performed a distinct crystallographic task. These programs were connected by binary files and the underlying operating system. The other major difference was that the user interacted with the programs via a question and answer dialog where reasonable defaults were suggested. The current approach is to provide as seamless a connection as possible from the data collection routines to NRCVAX. The programs have been repackaged so that as little user interaction as possible is required in routine situations. For more complex structures the full power of the system is still available. At the moment this is not an attempt at an automated structure determination package, rather it is assumed that a competent crystallographer is in control who can make intelligent decisions as required.

(1) Gabe, E.J., Le Page, Y., Charland, J.-P., Lee, F.L. and White, P.S. (1989) *J. Appl. Cryst.*, 22, 384-387.

Keywords: computer applications, single-crystal structure analysis, software design

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### Workflow and metadata in OLEX2

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Olex2 is an open source molecular graphics program [1] for solution, refinement and manipulation of small molecule crystal structures. There is an emphasis on usability and work-flow, which is achieved through a customisable and intuitive graphical-user-interface. The work-flow in Olex2 is designed to take a structure right from space group determination and solution through to refinement and preparation of the final structure report as simply as possible. Olex2 makes many complex crystallographic tools available to the user in a way that is intuitive for novice and experienced crystallographers alike. The program uses its own structure model, which can then be passed to structure solution and refinement programs such as ShelX. In addition, the program has its own structure solution and refinement methods based on the cctbx, which are available as a plug-in. Integration of the cctbx project with Olex2 is seen as key to future development of new and exciting features. The innovative history feature creates a history-tree for each individual solution of the structure, along with each stage of the refinement process. This enables the user to return transparently to an earlier point at any time, without having to concern themselves with saving and