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Structure solution of $Ag(pyz)_2S_2O_8$ in the presence of impurity phases using robust refinement

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High resolution powder diffraction was used to characterize a sample of $Ag(pyz)_2S_2O_8$. 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 Å b = 7.133 Å c = 14.586 Å β = 124.971°, 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[1] 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 χ^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 $Ag(pyz)_2$. The robustly refined structure will be presented, and compared to previous knowledge of this material.

[1] David, W. I. F., J. Appl. Cryst. (2001). 34, 691-698

Keywords: powder diffraction, robust refinement, structure solution

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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

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Applying parallel computing for a faster and better structure solution

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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

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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