MS31 O1

Obtaining integrated intensities for powder and granular materials Jon Wright *ESRF, BP-220, Grenoble 38043, France.* E-mail: wright@esrf.fr

Keywords: New powder diffraction techniques, powder and single crystal diffraction, Bragg intensity.

Integrated intensities which are extracted from powder data tend to be corrupted by the peak overlap problem. A variety of methods can be used to reduce this overlap problem by comparing several powder patterns. Eventually one has to guess how to partition the remaining overlaps in order to obtain a set of F(hkl) data. A likelihood based approach for dealing with this problem of overlapped powder data will be described [1]. The method has shown promising results in the application to severely overlapped data from powder protein samples.
Individual grains in powder specimens often give rise to diffraction spots when measured with a 2D area detector and microfocussed x-ray beam. Provided individual grains can be indexed, the problem of 2D spot overlap then replaces the problem of 1D peak overlap, and can be treated similarly.

[1] Wright, J. P., Markvardsen, A. J. and Margiolaki, I., *Z. Krist accepted.*

MS31 O2

Molecular envelopes from Protein Powder Diffraction Data C. Besnard [a], J. P. Wright [b], I. Margiolaki [b], S. Basso [a], F. Camus [a], A. N. Fitch [b], G. Fox [b], P. Pattison [a,c], M. Schiltz[a] [a] *Laboratoire de Cristallographie, École Polytechnique Fédérale de Lausanne (EPFL) Switzerland* [b] *European Synchrotron Radiation Facility (ESRF).* [c] *Swiss-Norwegian Beamlines (SNBL) at the ESRF.*
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Keywords: Powder Diffraction, Protein Structure Determination, Isomorphous Replacement.

The preparation of single crystals suitable for x-ray analysis is frequently the most difficult step in structural studies of proteins. If a microcrystalline powder sample can be obtained, de novo solution of the crystallographic phase problem can be achieved at low resolution via, for instance, the isomorphous replacement method. With synchrotron radiation and optimized instrumentation, high-quality powder patterns have been recorded from which it was possible to generate phase information for structure factors up to 6 Å resolution. pH- and radiation-induced anisotropic lattice changes were exploited to reduce the problem of overlapping reflections, which is a major challenge in protein powder diffraction. The resulting data were of sufficient quality to compute molecular envelopes of the protein molecule and to map out the solvent channels in the crystals, which are essential structural data for the characterization of microcrystalline proteins as novel mesoporous materials.

MS31 O3

In-situ neutron diffraction and gravimetric studies of H_2 cycling in hydrides William David[a,b], Marco Sommariva[a], Martin Jones[b], Simon Johnson[b] and Peter Edwards[b], [a]*ISIS Facility, Rutherford Appleton Laboratory, OX11 0QX, UK and* [b] *Inorganic Chemistry Laboratory, Unuiversity of Oxford, OX1 3QR, UK.*
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Keywords: hydrogen storage, neutron powder diffraction, gravimetric analysis

The safe and efficient storage of hydrogen is recognised as one of the key technological challenges in the transition towards a hydrogen-based energy economy. While hydrogen is stored at present principally using cryogenics or high-pressure, it is thought that the eventual solution may be a third way based upon novel condensed phase hydride stores. However, the multiple target criteria necessary for the successful implementation of such stores have not yet been met by any single material.
Simultaneously combining high storage capacity (>6wt% H_2) with a relatively low release temperature (<150^{o}C), complete reversibility of the thermal absorption/desorption cycle and also low toxicity / low cost is a difficult conundrum. All these scientific factors are intimately linked to the crystal structure of the hydrogen store and thus understanding hydrogenation requires a full structural description coupled with a detailed study of the physical and chemical properties of the absorption/desorption process of the system. To achieve this, we have recently developed a technique that allows us to perform simultaneous structural and gravimetric measurements on the GEM and HRPD diffractometers at the spallation neutron source, ISIS. The equipment, IGA^n, is based upon the Intelligent Gravimetric Analyser developed by Hiden Isochema Ltd. (UK).
This combined approach to the study of hydrogen absorption and desorption is extremely powerful; On the one hand, neutron diffraction, because of its sensitivity to hydrogen, is an ideal tool for the study of crystal structures of hydride while gravimetric analysis is an extremely precise tool for measuring mass change and thus monitoring hydrogen absorption and desorption under controlled temperature and pressure. We report here detailed absorption and desorption measurements on a number of systems that include the benchmark material Mg/MgD_2 and also the new Li_3N - Li_2ND - $LiND_2$ system. Our studies of Mg/MgD_2 confirm the consistency of results obtained from neutron diffraction and gravimetric analysis while our results on the Li_3N - Li_2ND - $LiND_2$ system reveal new mechanisms for both hydrogen absorption and desorption.

MS31 O4

Investigating Ionic Conductors by Powder Diffraction Hans Boysen, *Department of Earth and Environmental Sciences, Section Crystallography University of Munich, Germany.* E-mail: boysen@lmu.de

Keywords: ionic conductivity, neutron powder diffraction, diffusion

High ionic conductivity in solids plays an important role in technological applications such as in batteries, fuel cells, gas sensors, catalysts, etc. Valuable information to understand the underlying diffusion mechanisms can be gained from powder diffraction experiments, preferably with neutrons and under *in situ* (operating) conditions (high temperature, electric field). Analysis of the refined