

MS45-05 Structure determination by Multiple-wavelength anomalous dispersion (MAD) at the Pr LIII-edge Sandra Puehringer^{ab}, Michael Hellmig^a, Sunbin Liu^b, Manfred S. Weiss^a, Markus C. Wahl^b and Uwe Mueller^a
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The use of longer X-ray wavelengths in macromolecular crystallography has grown significantly over the past few years. The main reason for the increased use of longer wavelengths has been to utilize the anomalous signal from sulfur, providing a means for the experimental phasing of native proteins. Here we propose another possible application of longer X-ray wavelengths: MAD on the LIII-edges of various lanthanide compounds. A first experiment on the LIII-edge of Pr was conducted on the HZB MX beamline BL14.2, which resulted in the successful structure determination of the C-terminal domain of a spliceosomal protein. This experiment demonstrates that LIII-edges of lanthanides constitute potentially attractive targets for long wavelength MAD experiments.

Keywords: MAD phasing; lanthanides; long wavelength MAD;

MS46-01 The 2D-XPD charge flipping method for structure solution from powder diffraction data. Dan Xie^{ab}, Christian Baerlocher,^a Lynne B. McCusker,^a
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A single-crystal charge-flipping algorithm [1] has been applied to 2-dimensional projections derived from X-ray powder diffraction data to retrieve structure factor phases. These phases proved to be as reliable as those obtained from high-resolution transmission electron microscopy (HRTEM) images or from precession electron diffraction (PED) data [2]. The stronger reflections, in particular, tend to be correctly phased. The 2-dimensional electron density “images” obtained in this way show the same features as the corresponding HRTEM images. Application of the powder charge-flipping algorithm [3] to the full 3-dimensional powder diffraction data in conjunction with phases derived from several such (arbitrarily selected) projections was found to have a significant and beneficial effect on the structure solution. The approach was first developed and tested using data collected on the complex zeolites TNU-9 [4], IM-5 [5] and SSZ-74 [6]. All three of these structures were originally solved by combining X-ray powder diffraction and electron microscopy data, because X-ray diffraction data alone were not sufficient. In all three cases, the phase information derived from 2-dimensional subsets of the X-ray powder diffraction data resulted in a significant improvement in the electron density maps generated by the powder charge-flipping (pCF) algorithm. The inclusion of this phase information allowed all three structures to be determined from the X-ray powder diffraction data alone. Very recently, a true test of this 2D-XPD approach [7] was performed on the polycrystalline zeolite catalyst SSZ-82 [8], whose structure was not known and could not be solved by conventional pCF. Although most of the reflection intensities derived from the powder diffraction pattern were ambiguous because of reflection overlap (89%), they could still be used to retrieve reliable low-resolution phases in two dimensions. By imposing these few phases on the initial phase set for the 3D structure solution, interpretable maps could be generated. The framework structure, with 11 Si/B atoms in the asymmetric unit and a novel 12-/10-ring 2D channel system, could be seen clearly in the resulting electron density map [9]. This was not the case when the default mode of the pCF algorithm, starting with random phases, was used. This approach appears to offer a remarkably simple and powerful method for solving the structures of complex polycrystalline materials. The advantages of this 2D-XPD approach are two-fold: (1) only XPD data are required, so the more complicated electron microscopy experiments, whether HRTEM or PED, are not necessary, and (2) the projections used for phase retrieval can be selected arbitrarily. That is, a crystallite with the appropriate orientation and suitable thickness does not have to be found as it does for EM experiments. This 2D-XPD approach has the further advantage that it is general and can be applied to data collected on any polycrystalline material. No prior chemical knowledge other than the chemical composition is required.

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MS46-02 Neutron Scattering Studies of Ionically Conducting Oxides. Stephen Hull^a, Stefan T. Norberg^{ab}, Sten G. Eriksson^b.
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Solid Oxide Fuel Cells (SOFCs) are an important technology in the quest for more environmentally benign power generation. A key component within an SOFC is the solid electrolyte, which transfers O²⁻ ions from the air side (cathode) to the fuel side (anode) of the cell, with a compensating flow of electrons in the opposite direction through an external circuit which provides the electrical power. As a consequence, the material chosen for the role of the solid electrolyte must possess a high oxide-ion conductivity and low electronic conductivity (the latter essential to avoid internal short-circuits). At present, the most widely used material for the solid electrolyte in SOFCs is yttria stabilised zirconia (YSZ, Zr_{1-x}Y_xO_{2-x/2} with x typically around 0.15). However, these SOFCs must be operated at temperatures of ~1200K to achieve a sufficiently high ionic conductivity within the YSZ electrolyte. There are a number of advantages of SOFC operation at lower temperatures (~600-900K), including reduced problems of corrosion and shorter start-up times, which have motivated considerable research effort to identify materials with higher values of ionic conductivity than YSZ. These include the related material Zr_{1-x}Sc_xO_{2-x/2} and solid solutions based on Bi₂O₃ and CeO₂, which all possess an anion-deficient fluorite crystal structure (space group).

This presentation will briefly summarise our recent programme of neutron powder diffraction studies of the SOFC materials mentioned above, performed at the ISIS spallation neutron source. Particular emphasis will be devoted to the following aspects.

(i) The use of ‘total scattering’ methods (*i.e.* considering both the Bragg and diffuse scattering components), including reverse Monte Carlo analysis of the data to probe the nature of the defects within the materials.

(ii) The complimentary use of neutron powder diffraction and Molecular Dynamics simulations to investigate the nature of the ionic diffusion mechanisms and the factors which promote extensive oxide-ion diffusion.

(iii) The development of novel sample environment devices to allow neutron powder diffraction studies of SOFC electrolyte materials to be performed under conditions of temperature and gas atmosphere which mimic those found in real device applications.

Keywords: solid oxide fuel cells; neutron powder diffraction; reverse Monte Carlo