

FA3-MS04-O1

Opportunities for Observing Functional Materials in Operation Using Time-/Space-Resolved Diffraction. Paul Barnes. *Department of Chemistry, University College London, 20 Gordon Street, London WC1H 0AJ, U.K; and School of Crystallography, Birkbeck College, Malet Street, London WC1E 7HX, UK.*

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A full understanding of the working of functional materials requires the use of intense radiation sources to explore structure resolved in both time and space and within a working environment. The practical limits of measurement range in space from Å (atomic structure) to mm (real working systems) and in time from seconds (bulk solid state processes) upwards. *In situ X-ray diffraction* has emerged as a powerful technique for observing functional materials during both the synthesis stage and subsequently during their in-service performance. The revolution of the last two decades has been the realization of these goals using the unrivalled X-ray photon power made available through X-ray-dedicated synchrotrons. In this lecture I will give a personal account of this adventure, giving examples of “fast” *in situ* diffraction to follow rapid phase transformations or solid state reactions in response to changes in sample temperature, gas composition and pressure. This leads onto the use of energy-dispersive diffraction and the related *TEDDI* (Tomographic Energy-Dispersive Diffraction Imaging) technique for imaging of materials in action, such as catalysts, cements and functional oxides, and for exploring materials processes such as crystallization, transport and zonation. However the fuller objective, of imaging functional materials in *real time*, will require major advances in X-ray detection, and so a glimpse will be provided into current developments to make this happen.

Keywords: functional materials; space-resolved; synchrotron

FA3-MS04-O2

Kinetics of Solid State Reactions/Transitions Investigated by Real Time Neutron Spectroscopy. Kludia Hradil^a, Jeannis Leist^a, Friedrich Güthoff^a, Holger Gibhardt^a, Götz Eckold^a. *“Institut für Physiologische Chemie, Universität Göttingen, D-37077 Göttingen, Germany.*

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Inelastic neutron scattering technique provides information about the microscopic dynamics of solids. Investigations on a real time scale within external fields (temperature, pressure, magnetic/electrical fields) would yield the microscopic information of relaxation processes (phase transitions, domain order/disorder processes, decomposition processes).

Conventional inelastic neutron scattering technique is due to the low intensity of the investigated excitations and

therefore the relatively long measuring times beyond the scope for real-time experiments in timescales which are of interest. Eckold [1] introduced a method to combine real time resolution together with inelastic neutron scattering by a stroboscopic measuring technique. By cycling of the sample in an external field, the scattered intensity is not only detected as a function of momentum and energy transfer but also sorted within time channels. The reversibility of the processes is a necessary condition for the application of the technique. This technique was recently implemented within the spectrometer electronics of the triple axis spectrometer PUMA at FRM II and can provide the possibility to analyze excitations within relaxation processes on a timescale down to microseconds. Beside introducing the technique and possibilities for performing experiments first experiments applying electrical fields for the study of ferroelectrics or cycling of temperature to follow decomposition processes will be introduced.

[1] G. Eckold, *Nucl.Instr.&Methods*, **1990**, A289, 221.

Keywords: inelastic neutron scattering; in-situ experiments; time-resolved structural studies

FA3-MS04-O3

Time-resolved Binding of K₂PtBr₆ to Lysozyme by Protein Powder and Single Crystal X-ray. John Richard Helliwell^a, Tony A. Bell^b, Pat Bryant^c, Stu Fisher^{a,d}, George J. Habash^a, Madeleine Helliwell^a, Rena I. Margiolaki^e, Kaenket Surasak^a, Yves Watier^e, Jon Wright^e, Sampath Yalamanchilli^a. *“School of Chemistry, University of Manchester, UK. ^bSTFC Daresbury Laboratory, UK. ^cLife Sciences, University of Manchester, UK. ^dInstitut Laue Langevin, Grenoble, France. ^eESRF, Grenoble, France.*

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Protein powder diffraction continues to excite strong international interest; there are a variety of applications, including industrial protein characterization, such as polymorphs of insulin as well as extending structure determination to yet smaller crystal samples, which would otherwise be outside the range of synchrotron X-ray data collection from a protein microcrystal [1,2,3,4]. Furthermore there are the upcoming X-ray Laser Facilities, with possibilities for protein nanocluster-crystallites diffraction and de novo structure determination, as well as the MWatt spallation neutron sources for new neutron powder diffraction opportunities with neutrons as a neutral probe, for specific protein structural studies free of X-ray damage effects. Using ESRF ID31 we have recorded high quality protein powder diffractograms from K₂PtBr₆ bound to lysozyme at 80K to protect against X-radiation damage as much as possible and also to trap ie fix the K₂PtBr₆ heavy atom compound bound state. With multiple powder pattern analysis we have then extracted individual reflection intensities and thus been able to show the presence of PtBr₆²⁻ bound in lysozyme in (Fo-Fc) Fourier omit maps at two binding sites. The wavelength dispersive