8 01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

01.01 – Time Resolved Structural Studies

MS-01.01.01 TIME–RESOLVED STRUCTURAL STUDIES. By J.R. Helliwell, Department of Chemistry, University of Manchester, Manchester, M13 9PL, U.K.

Synchrotron radiation has considerably reduced the time taken to record data in; for example, X–ray crystallography, fibre diffraction, EXAFS, and powder diffraction. It has become more generally feasible therefore to perform time–resolved studies of molecules either of biological interest (e.g. enzymes and muscle) or in materials science during chemical reactions. An introduction to this topic will be given to open this microsymposium, which is organised under the auspices of the Commission on Synchrotron Radiation. A comparison will then be made of the currently available single crystal methods, particularly concerning exposure times and the elapsed times in acquiring a data set. This work shows that, even in X–ray crystallography of biological macromolecules, data sets can be rapidly recorded whilst of good quality and completeness.

MS-01.01.02 THE USE OF SYNCHROTRON RADIATION INSTRUMENTATION FOR TIME RESOLVED STRUCTURAL STUDIES: MUSCLE DIFFRACTION. By J. Bordas, N. Bliss, G. Diakun, C. Hall, J. Harries, R. Lewis, G. Mant, M. Martin-Fernandez, A. Svensson and E. Towns-Andrews*, SERC, Daresbury Laboratory, Warrington, U. K.

In recent years it has become evident that synchrotron radiation is essential for dynamical studies of biological structures. Biological specimens are inherently weak scatterers and hence time resolved measurements on systems of this type pose great demands on the experimental station design (e.g. station optics, data acquisition and detection systems). We are now able to collect two dimensional x-ray data in the millisecond time regime. Some data will be presented from time resolved diffraction studies of frog sartorius muscle. Experimental results and technical difficulties associated with such measurements, along with new instrumentation design, targeted specifically towards experiments of this nature, will be discussed. The layout of station 16.1, a high intensity fixed wavelength diffraction station at the SRS will be presented. This station will provide an increased x-ray flux at the specimen , approximately five times that of the existing time resolved diffraction station at the SRS. This will enable sub-millisecond data to be obtained and hence the possibility of following structural changes in biological systems in real time.

MS-01.01.03 TIME-RESOLVED X-RAY ABSORPTION SPECTROSCOPY by A.Fontaine.LURE (CNRS-CEA-MENC), Bât 209d F91405 Orsay -Cedex

X-ray Absorption Spectroscopy has proved to be a powerful tool to elucidate number of questions in materials science. Great interest exists in time-resolved experiments achieved with extreme energy resolution to take a full benefit of the strong correlation between the stereochemical environment of the absorbing atom and the exact shape and position of the absorption edge of the core levels. Time-resolved experiments based on XAFS invole generally mass transportation which means relatively slow processes. Selected experiments in electrochemistry are presented and different options to improve the time-resolution are rewieved.

Fast energy dispersive X-ray spectroscopy allows in-situ observations with data collected in a short time. A great benefit is expected from the forthcoming storage ring (ESRF) which should be able to give flux larger by at least 3 orders of magnitude. It is no longer possible to use the dispersive geometry to study samples at concentrations of about 100 µmol.They require a scanning monochromator, detecting the fluorescent decay. Related to this field are the quick-Exafs spectrometers developed initially at Hamburg under R.Frahm and at LURE under P.Lagarde, C.Prietto, H.Dexpert et M.Verdaguer which have been able in these three last years to open new routes in a wide variety of science. But the very first experiment to look for any kind of time-resolved X-ray Absorption Spectroscopy dealt with a stroboscopic approach.

MS-01.01.04 *IN SITU* DIFFRACTION EXPERIMENTS: RECENT ADVANCES AND RESULTS. By W. Depmeier, Inst. f. Mineralogie, Universität Kiel, D-W 2300 Kiel, Germany.

The scientific purpose of our group's *in situ* diffraction experiments is to follow structural changes of a given compound, observed by the changes of the corresponding diffraction pattern as a function of temperature, pressure, electron transfer rate, etc. The variables may be altered alone, or in combination. In pursuit of this idea our group has developed and built various sample environments for x-ray and neutron diffraction, and continues to do so. Such devices include

- a heatable high pressure cell for neutron powder diffraction (K. Fütterer)
- a microfurnace for use within a closed-cycle cryorefrigerator, allowing x-ray Guinier diffractometry at temperatures between 12 and, hopefully, 700 K (G. Hermeler)
- a heatable electrochemical reaction cell for *in situ* intercalation studies (H. Katzke).

The names in parentheses indicate the principal responsible scientist. Plans exist to further improve the instruments and to broaden the range of accessible variables.
Descriptions of the various sample environments will be presented and experiences discussed. Recent examples of results of our experiments will also be given. Materials studied include

- Li intercalated V_2O_5
- an ionic superconducting halide perovskite
- aluminate sodalites
- silica sodalites
- clathrasils.

MS-01.01.05A NEW TYPE OF DIFFRACTOMETER FOR QUICK DATA COLLECTION By Y. Ohashi,* H. Uekusa, A. Sekine and Y. Takenaka, Department of Chemistry, Tokyo Institute of Technology, Meguro-ku, Tokyo 152, Japan and T. Higashi and T. Sato, RIGAKU Corporation, Akishima, Tokyo 196, Japan

We found that the chiral alkyl group bonded to the cobalt atom in some cobaloxime complex crystals was racemized by X-ray or visible light without degradation of the crystallinity (Ohashi, *Acc. Chem. Res.*, 1988, **21**, 268-274). When the reactive alkyl group was replaced with more bulkier groups, the structural changes were so fast that the three-

