complexes involved in protein synthesis, recently we have expanded our studies to investigate structure and function of large eukaryotic multienzyme complexes such as the fatty acid synthase, a giant multifunctional enzyme that contains seven catalytic domains and catalyses all steps of fatty acid synthesis. We are using crystallography as the primary method in combination with electron microscopy and biochemical experiments.

Keywords: cellular processes, fatty acid synthesis, macromolecular assemblies

KN25.28

Acta Cryst. (2005). A**61**, C6

Microbeam Diffraction using High Energy Synchrotron Radiation

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The possibility of micron and sub-micron X-ray beams at High Energies (50-100keV) has opened up a wealth of new experimental possibilities. At the Materials Science beamline (ID11) of the European Synchrotron Radiation Facility (ESRF) a dedicated instrument, the three dimensional X-ray diffraction microscope (3DXRD), has been developed in collaboration with Risoe National Lab. Beam sizes ranging from 1 mm to 1 micron are available, and the combination of high flux and fast detectors allows for time resolved measurements. A number of examples will be presented to demonstrate the range of possible applications. The focus here will be on materials science applications, although these techniques are applicable to a large range of fields.

Among the applications presented will be the kinetics of recovery of sub-micron cells in highly deformed metals. The technique for constructing 3D maps of the full microstructure of materials (grain boundary morphology, grain orientation, elastic strain tensor) will be described, and a time resolved measurement of nucleation and growth of an imbedded grain within a highly deformed bulk metal will be shown in 4D.

Finally, the potential of achieving micron spatial resolution without micro focusing, that is using large beams, will be discussed with the obvious advantage of greater time resolution. Specifications for the current nanoscope project (an extension of the ID11 beamline due for completion in 2007) will be briefly described.

Keywords: microbeams, X-ray diffraction, materials science

KN26.28

Acta Cryst. (2005). A**61**, C6

New Possibilities for Structure Determination of Biomolecular Complexes

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New possibilities to analyse the structure of macromolecular complexes using small-angle scattering (SAS) of X-rays and neutrons are presented. SAS allows one to study the overall structure of native particles in solutions and to analyse structural changes in response to variations in external conditions. Recent progress in instrumentation and data analysis [1] significantly enhanced resolution and reliability of structural models provided by the technique and made SAXS a useful complementary tool to high resolution methods, especially powerful in the analysis of complex macromolecules. The latter mediate most of fundamental biological processes and the focus of modern structural biology is rapidly shifting towards their study.

Advanced approaches to analyze macromolecular complexes in solution using SAS will be presented including: *ab initio* low resolution structure analysis, rigid body refinement and addition of missing fragments to high resolution models, analysis of equilibrium mixtures and the use of contrast variation and specific deuteration in neutron SAS Practical applications of the methods will be illustrated by recent examples.

[1] Svergun D. I., Koch M. H. J., *Rep. Progr. Phys.*, 2003, **66**, 1735.

Keywords: small-angle scattering, rigid-body analysis, biomacromolecular structures

KN27.29

Acta Cryst. (2005). A**61**, C6

Bound Ligands to Probe the Activity of Type 2 Copper Sites in Proteins

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Nitrite reductase (NiR) is a type 2 copper-containing enzyme that reduces nitrite to nitric oxide as part of the global nitrogen cycle. Type 2 copper sites are found in a variety of versatile oxidoreductases that mediate reactions involving oxygen or nitrogen oxides and are found throughout all branches of life. Crystal structures of (NiR) to beyond 1.4 Å resolution with bound nitrite and nitric oxide have given insight into the catalytic mechanism which differs from that of heme cd_1 NiR. Mutagenesis studies of copper NiRs show that an aspartate – histidine pair in the active site is found to control binding of copper ligands and largely define the chemical reactivity of NiR. Unexpectedly, nitrite and nitric oxide are bound in an almost face-on and side-on coordination to the copper. In contrast, the inhibitor azide binds end-on to the type 2 copper. Also, acetate and nitrate coordinate through both oxygens (bidentate), whereas nitrite is coordinated by a single oxygen that forms an H-bond to the active site aspartate, an interaction that is likely to be essential for efficient catalysis.

Interestingly, NiR is able to reduce oxygen to hydrogen peroxide in vitro, eventually leading to enzyme inactivation. The NiR type 2 copper site shares a similar coordination sphere to those of superoxide dismutase and amine oxidases suggesting the possibility of common mechanistic features with respect to reactivity of these sites with oxygen and nitrogen oxides.

Keywords: structures of metalloproteins, copper complexes, nitric oxide

KN28.29

Acta Cryst. (2005). A**61**, C6

Crystallochemical Basis of Synthetic Mineral Immobilisation

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Designing contemporary methods for immobilising pollutants is underpinned by crystal chemical and mineralogical principles [1], with the selection of a ceramic or synthetic mineral immobilisation matrix governed by several considerations. First, toxic metals should be incorporated in their least toxic chemical states. For example, substances that eschew As^{3+} and Cr^{6+} in favour of less dangerous As^{5+} and Cr^{3+} are preferable. Second, as this illustration indicates, it is sometimes necessary to simultaneously accommodate the oxidized and reduced species of different metals, restricting the suite of mutually compatible minerals that can be selected. Third, crystal structures are preferred that have multiple cation and/or anion acceptor sites as this minimizes the number of phases required to crystallise simultaneously, and allows greater flexibility to respond to variations in waste stream composition. This in turn limits the chance of undesirable compounds forming. Finally, phases with large numbers of appropriate cation acceptor sites are advantageous, as they result in higher waste loadings and less 'bulking' of the waste product through the introduction of inert additives. These matters will be illustrated by reference to the zirconolites [2], already being used for nuclear waste treatment, and the apatites [3,4] that are potential materials for the fixation of hazardous waste.

[1] Haggerty S.E., *Ann. Rev. Earth Planet. Sci*.,1983, **11**, 133. [2] Grey I.E., Mumme W.G., Ness T.J., Roth R.S., Smith K.L., *J. Solid State Chem.*, 2003, **174**, 285. [3] Ioannidis T.A., Zouboulis A.I., *J. Hazardous Materials*, 2003, **B97**, 173. [4] Dong Z.L, White T.J., *Acta Crystallogr.*, 2004, B**60**, 138.

Keywords: waste management, environmental chemistry, environmental affairs