for phase extension and refinement of main reflections, and then used for phase extension from main reflections to satellite reflections.

[1] De Wolff P.M., *Acta Cryst. A*, 1974, **A30**, 777. [2] Hao Q., Liu Y.W., Fan H.F., *Acta Cryst*. 1987, **A43**, 820.

Keywords: incommensurate modulated structure, high-resolution electron microscopy, electron diffraction

MS91.30.4

Acta Cryst. (2005). A**61**, C116

Electron Dynamical Diffraction Imaging and Diffuse Scattering by Small Dislocation Loops

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Effects of dynamical scattering of high-energy electrons by elastic fields of interstitial or vacancy loops in a crystalline material provide a convenient means for diffraction contrast imaging. In this presentation we describe new developments in the methodology of simulation of diffraction images and *dynamical* diffuse scattering by small dislocation loops. To simulate diffraction images, a many-beam Howie-Basinki equation approach has been developed where strong dynamical effects as well as the non-parallel propagation of diffracted beams in the crystal are treated using a combination of the adaptive spatial mesh and wave field interpolation techniques. The significance of dynamical diffraction as well as practical applications of the new approach are illustrated by the comparison of simulated and experimentally observed images. The treatment of diffuse scattering includes effects of Kikuchi diffraction on Huang diffuse scattering patterns that we simulate using the atomic displacement fields evaluated using anisotropic elasticity solutions and atomistic modelling.

Keywords: electron microscopy and diffraction, quantitative electron diffraction, dynamical diffraction

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Characterization of Nanophases in HRTEM: Fourier Transform and Simulation

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High resolution transmission electron microscopy (HRTEM) was applied to study the microstructure of biomaterials based on calcium phosphates: α-tricalcium phosphate, octacalcium phosphate (OCP) and hydroxyapatite (HAP). Phase analysis at nanolevel was required to evaluate whether the final product included one or several Ca phosphate modifications. Due to high sensitivity of all these compounds to irradiation of the convergent electron beam such local analysis was performed by processing diffractograms (Fourier transform) from HRTEM images with Digital Micrograph software (Gatan). Interpretation of the experimental results was done by the means of simulation of selected area electron diffraction patterns and HRTEM images using JEMS [1], which allows to perform large calculations of dynamical diffraction patterns and HRTEM images for big multiatomic crystallographic unit cells.

HAP nanocrystals (5-20 nm) randomly oriented relatively to each other were identified in plasma sprayed coatings on different substrates. OCP crystals were found to contain HAP inclusions and their sizes were dependent on crystal growth regime. Phase transformation during high temperature synthesis of α-tricalcium phosphate from the β-form has been studied.

[1] JEMS: http://cimewww.epfl.ch/people/Stadelmann/jemsWebSite/jems.html Stadelmann P..

Keywords: electron microscopy and diffraction, simulation, calcium compounds

MS92 EMERGING TECHNOLOGIES FOR STRUCTURAL BIOLOGY

Chairpersons: Sine Larsen, Michael Becker

MS92.30.1

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Diffraction from a Laser-aligned Beam of Hydrated Proteins

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The aim of this work is to solve proteins which cannot be crystallized. An apparatus is under construction at ASU physics (electrons) and at the Advanced Light Source in Berkeley (X-rays) to obtain diffraction patterns from a single-file submicron droplet stream [1]. Each water droplet contains, on average, one protein. The droplets freeze by evaporative cooling to vitreous ice, most of which is allowed to sublimate. The molecules are aligned by a 100 watt CW fiber laser. All three beams , laser, X-rays and droplets, run continuously, and diffraction data is aquired continuously by CCD camera until adequate signal-to-noise is achieved. The laser polarization is then rotated into a new orientation using a quarter-wave plate, allowing tomographic diffraction data collection for three-dimensional reconstruction. The phase problem for the continuous diffraction pattern is solved by novel iterative Gerchberg-Saxton-Fienup methods [2]. Waves scattered by different molecules don't interfere. The requirements of laser power and droplet temperature needed to achieve sub-nanometer resolution and so observe the secondary structure of proteins will be described in detail. Factors which affect the damping of oscillations in the laser beam and momentum transfer by elastic diffraction to a levitated molecule.

[1] Spence J., Doak B., *Phys. Rev Letts*, 2004, **98**, 198102. [2] Spence J. et al, *Acta Cryst A*, 2005, in press. [3] Marchesini S. et al., *Phys Rev.*, 2003, **B68**, 140101(R).

Keywords: proteins, structure, laser alignment

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Protein Structures without Crystals

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Many proteins are inherently difficult to crystallize, due to various physical properties, e.g. membrane proteins, due to their large hydrophobic area, or amyloid-forming peptides and proteins due to very strong hydrogen bond networks in combination with hydrophobic interactions. Novel light sources (X-ray free electron lasers) may enable us to obtain structural information from small non-crystalline samples if we are able to gather enough scattering data before radiation damage destroys the sample. On the other end of the spectrum computer simulations of molecular dynamics may be able to predict structures of small proteins based on force fields within the near future. In the current presentation I give an overview of our work in both areas and how they are connected.

Keywords: X-ray, fel, gromacs

MS92.30.3

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Structural Proteomics using NMR in RIKEN Structural Genomics/Proteomics Initiative

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RIKEN Structural Genomics/Proteomics Initiative (RSGI) (http://www.rsgi.riken.jp) was organized by RIKEN Genomics Sciences Center and Harima Institute at SPring-8 in 2001. RSGI has been integrated into the National Project on Protein Structural and Functional Analyses ("NPPSFA" or "Protein 3000"), organized by the Ministry of Education, Culture, Sports, Science, and Technology (MEXT), as one center of the program for comprehensive studies. We are now focusing on proteins involved in phenomena of biological and

medical importance. Both NMR spectroscopy and X-ray crystallography are used for protein structure determination. To accelerate the NMR analysis, we have constructed the large-scale NMR facility housing 40 high-field NMR spectrometers, and developed several key technologies such as a high-yield cell-free protein synthesis system for high-throughput and automated production, a software package, KUJIRA, for the systematic and interactive NMR data analysis, and the program CYANA for automated structure calculation. We determined 75 structures in 2002 fiscal year, and 207 structures in 2003 fiscal year, respectively, by NMR spectroscopy.

Keywords: NMR spectroscopy, structural proteomics, cell-free protein synthesis

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X-ray Microscopy Project at NSRRC

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Very recently, under the NSRRC X-ray Microscopy Project, we have installed a transmission X-ray microscope (TXM) to the BL01B end station of an advanced, high flux (3×10^{11} photon/s) and wide energy spectrum (7-23 keV), X-ray source generated by a superconducting wavelength shifter. The state-of-art TXM can provide 2D imaging and 3D tomography for imaging light materials such as biological specimens with a spatial resolution of 30-60 nm, using the Zernike-phase contrast capability with 8-11 keV hard X-ray. To our best knowledge, such resolution achieved is unprecedented in X-ray imaging up to date. In this presentation, we would like to share the scope and the prospective of the project as well as the progress of the TXM in our center. The impact of our TXM is expected in many imaging works for buried structures, including the analysis of failure mechanisms in microelectronic devices due to electromigration, thermal breakdown or inhomogeneity, or the characterization of porous materials such as soils and rock, and the transportation behavior in these porous structures. In addition, material failures due to induced strain, crack propagation or corrosion can be studied with our modern X-ray microscope of 2D and 3D imaging capability. Currently, we aim our unprecedented X-ray microscope at the research of cells in life science. With the 3-D "virtual sectioning" capacity to be matured, we intend to view either a single cell, cell clusters, or any region of a tissue. With labeling agents, for instance, gold, for contrast variation, in-situ imaging for specific cellular functions is possible with our TXM.

Keywords: X-ray imaging, X-ray microscopy, in-situ imaging

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Analysis of Liquid and Crystalline Proteins by Particle Induced X-ray Emission (PIXE)

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Unique identification of *metals* bound to macromolecules is an interesting challenge in structural biology, and an unambiguous assignment is often problematic. microPIXE (particle induced X-ray emission) with 2-3MeV protons on liquid and crystalline proteins has been used very successfully in both identifying elements and in measuring their stoichiometric ratio (calibrated per protein molecule by using the sulphur peak to give an internal normalisation of the sulphur atoms from the known cysteines and methioniones) to an accuracy of between 10 and 20% on over 50 samples [1,2].

Measurements using the technique have informed a wide range of questions, including the degree of seleno-methionine incorporation into a proteins destined for MAD structure determination, the identity of unexpected electron density in solved structures, identifying of metals bound to liquid protein samples to elucidate their function prior to structural studies, determining whether or not DNA is bound to a protein crystal (from the phosphorus to sulphur ratio), checking for paramagnetic species in proteins prior to NMR analysis, and analysing proteins before and after mutation of putative metal binding sites.

The method is now routine and may have potential as a high throughput screening tool in structural biology.

[1] Garman E., *Structure*, 1999, **7**, R291-R299. [2] Garman E.F., Grime G.W., *Progress in Biophysics and Molecular Biology*, 2005, *in press*.

Keywords: PIXE, proteins, trace-metal analysis

MS93 CRYSTALLOGRAPHY AND ENVIRONMENTAL SCIENCE

Chairpersons: Marcello Mellini, Mihaly Posfai

MS93.30.1

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Application of Natural Zeolites: Understanding the Properties at a Molecular Scale

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Natural zeolites are usually found as zeolite-rich rocks (zeolitites) which contain at least 50 wt% of zeolite phase. Italian zeolitites may contain phillipsite or chabazite with an overall content of zeolite phase as large as 70 wt%. Especially for agronomical and agricultural purposes, an important property is the adsorption and/or release of the ammonium ion. In this frame, the aim of this study is to present the structures of NH_4 exchanged chabazite and phillipsite and to explain the different behaviour of the two zeolites in agronomy and agriculture applications. It is shown that the knowledge of the local environment of NH_4^+ in the cavities of these zeolite species is extremely important. In chabazite, the ammonium ion with a monodentate local structural environment may be easily released or desorbed. NH_4-phillipsite [1] shows instead that the ammonium ion is in a tridentate local environment and it is consequently more difficult to be released or desorbed in solution. As a matter of fact, the zeolitite with NH_4-exchanged chabazite gave very encouraging results in agronomy applications. On the contrary, the zeolitite with NH_4-exchanged phillipsite gave very poor results for the same application [2].

[1] Gualtieri A.F., *Acta Cryst*., 2000, **B56**, 584. [2] Mazzocchi R., Casalicchio G., Giorgioni M.E., Loschi B., Passaglia E., Savelli C., *Colture Protette*, 1996, **11**, 91.

Keywords: natural zeolites, ammonium ion, application

MS93.30.2

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A Structural View of Carbonate Biomineralization by Bacteria

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Although it has been recognized for more than a century, biomineralization of carbonate minerals by prokaryotic organisms has been much less studied, from a structural point of view, than the formation of calcite and aragonite by eukaryotic cells. Formation of carbonates by bacteria and archaea has however a potential strong environmental significance, for example for immobilization of radionuclides under aridic conditions or for deep geological carbon dioxide mineral sequestration.

Investigation tools such as analytical transmission electron microscopy and synchrotron-based scanning transmission x- ray microscopy have allowed us to evidence, at nanometer scale, the well known, yet poorly understood, systematic relationship between bacterial extra-cellular polysaccharides and carbonates. We report examples from mineral (pyroxene) surface micro-habitats and from lacustrine carbonate microbialithes. Nanobacterial-like morphologies are characteristic of these carbonate crystals, the formation mechanism of which will be discussed.

A second mode of carbonate and phosphate biomineralization by bacteria has also been evidenced. It is radically different in that it involves intracellular, particularly periplasmic, components. Possible