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

MS92.30.4

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X-ray Microscopy Project at NSRRC

Mau-Tsu Tang, Yen-Fang Song, Gung-Chian Yin, Te-Hui Lee, Cheng-Hao Ko, King-Long Tsang, Keng S. Liang, *National Synchrotron Radiation Research Center, Hsinchu 30076, Taiwan.* E-mail: mautsu@nsrrc.org.tw

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)

Elspeth F. Garman[a], Geoff W. Grime[b], [a]*Department of Biochemistry, University of Oxford, U.K.* . [b]*University of Surrey, Department of Physics, Guildford GU2 7XH, U.K.* . E-mail: elspeth@biop.ox.ac.uk

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.

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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

Alessandro F. Gualtieri, Elio Passaglia, *Department of Earth Sciences, University of Modena and R.E., Modena, Italy.* E-mail: alex@unimore.it

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].

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Keywords: natural zeolites, ammonium ion, application

MS93.30.2

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A Structural View of Carbonate Biomineralization by Bacteria

François Guyot, Karim Benzerara, Nicolas Menguy, *Institut de Minéralogie et de PMC et Institut de Physique du Globe de Paris, France.* E-mail: guyot@lmcp.jussieu.fr

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

mechanisms implying periplasmic protein activity (e.g. alkaline phosphatase) and metabolic activity (e.g. sulfate reducing bacteria) will be reviewed.

Keywords: biomineralization, carbonate formation, polysaccharides

MS93.30.3

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Nano-scale Studies of Processes on Crystal Surfaces in Aqueous Solutions

Guntram Jordan[a], Kirill Aldushin[a], Wolfgang W. Schmahl[b], [a]*Inst. f. Geol., Mineral. u. Geophys., Ruhr-Universität, Bochum, Germany.* [b]*Dept. f. Geo- u. Umweltwiss., LMU, München, Germany.* E-mail: guntram.jordan@rub.de

At the crystal-water interface a large diversity of processes takes place which influence or even control environmental conditions. Among these processes are sorption, growth, dissolution, formation of surface complexes or metastable phases by leaching, repolymerization, or precipitation. For a detailed understanding of these processes, factors and properties such as the stability of metastable phases or structural frameworks need to be taken into account.

Hydrothermal atomic force microscopy has been used for nano-scale in-situ investigations of crystal surfaces in aqueous solutions [1-3]. The method can provide insights into the molecular mechanisms and kinetics of solid-liquid interface processes. The results stress that especially for processes taking place at silicate-water interfaces the consideration of the stability of metastable states and structural influences is very important. In contrast, mechanisms of processes at interfaces like the carbonate-water interface although largely unsolved rather seem to comprise sequences of less numerous steps.

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Keywords: surfaces and interfaces, AFM, silicates

MS93.30.4

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Hydrothermal Preparation of TiO2: AC Composite Crystalline Particulates

A. K. Subramani[1], K. Byrappa[1], R. Dinesh[2], K. M. L. Rai[3], S. Ananda[3], M. Yoshimura[2], [1]*Departmant of Geology, University of Mysore, Manasagangotri, Mysore –6.* [2]*Materials Research Laboratory, Tokyo Institute of Technology, Nagatsuta, Midori, Yokohoma - 226, Japan.* [3]*Departmant of Chemistry, University of Mysore, Manasagangotri, Mysore –6.* E-mail: byrappak@yahoo.com

A highly active, monodispersed designer crystalline nano-particulate TiO_2 has been impregnated onto the activated carbon surface under mild hydrothermal conditions (<250^{0}C, P~40 bars) which finds the application as photocatalyst. Conventionally TiO_2 is prepared through solid state reactions, etc; further the hydrothermal impregnation of such particulates onto the surface layers of activated carbon has not been carried out either to. The hydrothermal technique provides an easy and one-step method to obtain monodispersed and well crystallized desired products and also eliminates the high temperature firing or pyrolysis required by the other methods. In the present study various hydrothermal experimental parameters like the starting precursors, mineralizers, temperature, etc., were taken into consideration for the impregnation experiments. The as-prepared catalyst composite was characterized by various techniques like XRD, SEM-EDX, PALS, BET and FTIR. The XRD results showed the persisting nature of anatase phase of TiO_2 deposited on the activated carbon surface. The BET and FTIR results reveal an optimum (TiO_2 to AC ratio) conditions for the impregnation. The PALS results further confirmed that TiO_2 is impregnated onto the surface and wider pores (macro- and mesopores) of the activated carbon and the micropores do not play a significant role as far as the TiO_2 impregnation is concerned. The results of the study finally revealed that TiO_2 could be effectively impregnated onto the activated carbon surface layers under mild hydrothermal conditions and such a designer crystalline particulate composite is highly useful for the environmental issues such as degradation of hazardous organics/wastes, treatment of effluents, air purification and so on.

Keywords: hydrothermal impregnation, photocatalyst, TiO_2: AC composite

MS93.30.5

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Crystalline Structure of Biodegradable Polyhydroxybutyrate thin Films

Katsuhito Mori[a], Harumi Sato[a], Hikaru Terauchi[a], Isao Takahashi[a], Yukihiro Ozaki[a], Isao Noda[b], [a]*School of Science and Technology, Kwansei Gakuin University, Japan.* [b]*The Procter and Gamble Company, U.S.A.* E-mail: scbc0010@ksc.kwansei.ac.jp

Polyhydroxybutyrate: PHB and random copolymer, Polyhydroxyalkanoates: PHAs are crystalline biodegradable polyesters. As a substitute for petrochemical materials, the study of biodegradable polymer has attracted considerable attention. Our recent study demonstrated that melting behavior of a new random copolymer, Poly(3-hydroxybutyrate-co-3-hydroxyhexanoate): P(3HB-co-3HHx) showed a sharp contrast with that of PHB. A novel intermolecular interaction successfully explained the results.

As a next step, we are now conducting the X-ray reflectivity (XR) and grazing incidence X-ray diffraction (GIXD) measurements of thin films of PHB and P(3HB-co-3HHx) at various temperatures. The aim of this study is to get information on morphology, crystallinity, and crystal structure in the surface and thin films, which must be crucial for understanding the physical properties peculiar to the surface region and the mechanism of bio-degradation on a microscopic standpoint.

Both PHB and P(3HB-co-3HHx) thin films indicated that the crystallites tend to orient their *b*-axis along the surface normal direction. The present results strongly support the intermolecular interaction along the *a*-axis direction, which was suggested by the previous study on bulk samples. According to Bragg reflection from the near-surface region, surface morphology of PHB is different from that of P(3HB-co-3HHx) even at room temperature. We will also discuss the results of FT-IR spectrum obtained from the thin films.

Keywords: biodegradable polymer, X-ray diffraction, thin film

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MS94 CRYSTALLOGRAPHIC KNOWLEDGE IN DRUG DESIGN STRATEGIES

Chairpersons: Franck Leveiller, Michele Saviano

MS94.30.1

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Can Structures lead to Better Drugs? Lessons from Ribosomal Antibiotics

Ada Yonath, *Structural Biology Department, Weizmann Institute.* E-mail: ada.yonath@weizmann.ac.il

Ribosomes, the universal cellular organelles catalyzing the translation of genetic code into proteins, are giant asymmetric riboprotein assemblies with a striking architecture and inherent mobility, enabling their function as ribozymes how place their substrate in stereochemistry suitable for peptide bond formation and substrate mediated catalysis. As the main player in a fundamental cell process, ribosomes are targeted by many antibiotics. Structures of over a dozen antibiotics complexes, obtained by using eubacterial ribosomes suitable to serve as pathogen models at clinically relevant concentrations, showed that although theoretically the giant ribosome offers numerous binding opportunities, ribosomal antibiotics bind to a single or a few binding sites; that most antibiotics interact primarily with riboso mal RNA and cause minor conformational changes; that minute structural differences, scattered in various ribosomal locations, are responsible for antibiotic selectivity; that the properties of the antibiotic-binding modes are dictated by species-specific binding pocket composition and conformation, the functional state of the ribosome, and the drugs chemical nature; that resistance to ribosomal