

ESRF Users' Meeting 2009: reports from parallel sessions

MATERIALS SCIENCE PARALLEL SESSION

Organisers and Chairs: Chiara Maurizio (Users Organization & Italian CRG), Andy Fitch (ESRF)

The Materials Science Parallel Session was attended by about 30 people. It started with brief talks given by the scientists in charge of the beamlines that are part of the Materials Science group, that presented the improvements and the new instrumentation installed during the last year; the beamlines concerned were ID09A, ID09B, ID11, ID15, ID27, ID31 and the new ID06, dedicated to high pressure experiments with a large volume press, that should welcome the first users in Nov.-Dec. 2009. Then Andy Fitch leaded a discussion on the data archiving, presenting the recommendations from the ESRFI working group about digital repositories: in the future, all raw data from the experiments produced with public funding should be available and open access, together with all the suitable correlated information needed to analyze them. Then, in the framework of the ESRF upgrade programme, the proposals for two new beamlines (UPBL2 and UPBL9) were presented. This first section of the parallel session was so rich that it took half an hour more than what foreseen.

The second part of the parallel session was then dedicated to four scientific presentations that, as commonly encountered in the materials science field, covered quite different subjects. The first one was about morphological clues to explain the wet granular pile stability and it was given by Ralf Seemann (Max Plank Institute, Germany): he showed that the fact that the mechanical properties of a wet granular pile are quite insensitive to the liquid content is due to the specific organization of the liquid in the pile into open structures. The second presentation, given by Alberta Bonanni (University of Linz, Austria) was about diluted magnetic semiconductors for spintronics, where synchrotron radiation techniques such as x-ray diffraction and absorption allowed investigating the aggregation of the magnetic cations in these materials, that strongly determines the magnetic properties. The third talk was given by Qingyu Kong (Synchrotron SOLEIL, France), that presented how the picosecond x-ray scattering can resolve the molecular geometries of short-lived transient intermediates and free radicals in a chemical reaction. Finally, Paul Raterron (CNRS, France) showed how the rheology of Earth materials can be investigated by large-volume high-pressure setups. Each talk was followed by several questions asked by the audience that could enter in the different subjects thanks to the efficacy and clarity of the contributions presented.

HIGH RESOLUTION AND RESONANCE SCATTERING SESSION

Organisers and Chairs: Tullio Scopigno (Users Organisation & University of Rome "Sapienza", Italy), Rudolf Rüffer (ESRF)

ESRF CONTROL ROOM MEETING ROOM 4 February 2009

The parallel session started regularly at 14.00 and there were four presentation:

Lattice dynamics in elemental metals with incommensurate crystal structures, Ingo Loa, University of Edinburgh, Edinburgh, UK

High pressure/high temperature: A view into the deep Earth, Catherine McCammon, Universität Bayreuth, Bayreuth, Germany

Magnetic Circular Dichroism effects in the Resonant Inelastic X-ray Scattering at the K (pre) edge of 3d transition metal ions: an outlook, Amélie Juhin, University of Utrecht, Utrecht, the Netherlands

Fine details of cobalt 1s pre-edges revealed by RXES, Gyorgy Vanko, KFKI Research Institute for Particle and Nuclear Physics, Budapest, Hungary

The parallel session was attended by nearly 30 people.

MACROMOLECULAR CRYSTALLOGRAPHY SESSION

Organisers and Chairs: Gerlind Sulzenbacher (User Organisation & CNRS-Univ. Marseille), Sean McSweeney (ESRF)

The Macromolecular Crystallography (MX) parallel session was held at ILL Chadwick Amphitheatre and was attended by about 60 people. About twenty participants were students attending the MX-School “Getting the most from the ESRF macromolecular crystallography beam-lines”. The parallel session focused on the principle challenges of Structural Biology in the future and how these challenges can be addressed by Instrument and Methodology developments.

Sean McSweeney, ESRF, started the session with a brief overview of MX within the ESRF Upgrade. His presentation led over to a talk by Matthew W. Bowler, ESRF, on the next generation of MX sample evaluation. The following part of the session was dedicated to data collection with microcrystals. Christoph Müller-Dieckmann, ESRF, gave an overview of what can be achieved by microcrystallography, of the major problems related to the technique, and future developments as part of the future plans of the ESRF Upgrade. Patricia Edwards, M.R.C. Laboratory of Molecular Biology, Cambridge, UK, explained how the use of ESRF beam-lines allowed the structure solution of several G-protein coupled receptors, including the first structure of a beta-1-adrenergic receptor. Her results illustrated further new opportunities for solving structures from much smaller crystals than previously anticipated. After this microcrystal interlude Don Jorge Navaza, IBS, LMES, Grenoble, explained why low resolution data are important and how they can fill the gap between crystallography and electron microscopy. The last talk of this very charged session was given by Rudolph Dimper, ESRF, and he presented a panel of reflections on Data Management. This last part illustrated the problems related to the archiving of huge amounts of data and the solutions contemplated by the ESRF computing group.

This MX parallel session had a very dense programme of presentations and very little time was left for discussion, which focused mainly on methodology.

X-RAY IMAGING SESSION

Organisers and Chairs: Eric Maire (User Organisation & INSA, Lyon, France), José Baruchel (ESRF)

The parallel session of imaging gathered about 40 attendees during the two hours allocated for the session. Three scientific talks were presented by J. Cambedouzou, P. Bleuet and A. King. The three authors exposed results of their team in new interesting topics in the field of imaging at the ESRF. The hot topics this year are nano imaging, diffraction imaging and fluorescence. The session also included a discussion on the upgrade, introduced by a presentation of the status by J Baruchel, and a discussion on data storage at the ESRF, introduced by T. Weitkamp.

14:00 - 14:10 A report on MIA-NiNA and clinical trials
José Baruchel, ESRF, Grenoble, France

14:10 - 14:40 Diffraction contrast tomography - 3D orientation mapping in polycrystals
Andrew King, ESRF, Grenoble, France and University of Manchester, UK

14:40 - 15:10 Hard x-ray fluorescence and diffraction scanning tomography
Pierre Bleuet, CEA and ESRF, Grenoble, France

15:10 - 15:40 Carbon nanotubes interaction with macrophages: chemical imaging by synchrotron X-ray fluorescence microscopy
Julien Cambedouzou, Université Paris-Sud, CNRS, Orsay, France

15:40 - 16:00 Discussion
ESRF Upgrade Programme and data archiving at the ESRF

SURFACES AND INTERFACES SCIENCE SESSION

Organisers and Chairs: Christian Kumpf (User Organisation & Research Center Jülich, Germany), Jörg Zegenhagen (ESRF)

The parallel session on “Surfaces and Interfaces Science” was again a very lively event with approximately 50 people attending. As usual the program consisted of three ESRF-internal and three external contributions which were all intensively discussed by the audience.

The session started with a contribution by Thomas Cornelius on present and future developments at ID 01 with a focus on micro X-ray diffraction. He showed data from a combined XRD and AFM study: Crystal truncation rods from SiGe islands were recorded while mechanical pressure was put on the sample by an AFM tip. In the second contribution Olivier Balmes gave an overview on new detector systems (MAXIPIX and MAR) and several sample environments, which are now available at ID 03. He also presented in-situ surface diffraction results obtained from investigations on catalytic CO oxidation in a flow reactor. The “internal” part of the session was then closed by Blanka Detlefs reporting the progress in commissioning the new end station for hard X-ray photoelectron spectroscopy (HAXPES) at ID 32. The new machine will have a resolution of 160 meV@6.5 keV. Results from the first user experiments on $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ / La_2CuO_4 were also shown.

In the second part of the SIS parallel session three contributions from external users were presented: At first Holger L. Meyerheim (MPI für Mikrostrukturphysik Halle) showed his interesting results on Co nanoislands formed on a Cu(001) surface. In a detailed surface x-ray diffraction (SXRD) study the relaxation behavior of the Cu substrate due to the island-induced stress was studied. The second talk was given by Geoff Thornton from the University College London. He reported on the properties of the $\text{TiO}_2(110)$ and (011) surfaces regarding photo catalysis. Different models based on SXRD and density functional theory (DFT) were discussed for the oxygen-terminated surfaces. Finally Julian Stangl (Johannes Kepler University, Linz) showed his results on semiconductor nanostructures obtained with a focused x-ray beam. By scanning the surface with a 100 nm beam individual (self assembled) nanostructures were investigated in terms of strain state and plastic relaxation. Exemplary data for SiGe nano-islands and InAs nano-wires were presented.

SOFT CONDENSED MATTER SESSION

Organisers and Chairs: Stephan Roth (User Organisation & HASYLAB Hamburg, Germany), Theyencheri Narayanan (ESRF)

The soft condensed matter parallel session was focused on two areas, namely the partnership for soft condensed matter, and scientific highlights from the SCM beamlines presented by a total of three invited speakers from the SCM user community.

The session (programme see below) started with a report on the partnership for soft condensed matter by T. Narayanan (ESRF). Based on the core techniques SAS, GISAS, GID, XPCS and NSE, the center is proposed to include ILL and ESRF SCM beamlines. He explained the idea of joint neutron and synchrotron beamtime especially in the field. The laboratory equipment is foreseen to provide complementary techniques, such as Brewster-angle spectrometers, rheometers, light scattering as well as sample preparation capabilities. It should provide a key platform for industrial and applied research. The important point is that its implementation must give an added value for both users *and* staff of ESRF.

In the first scientific lecture O. Francesangeli reported on the supramolecular structure and phase behavior of selfassembled liposome-DNA-metal complexes for gene transfer with future applications as non-viral in gene therapy. In detail, he presented recent synchrotron XRD measurements performed at ID02 at low concentration of the PEG-lipid component have shown the self-assembled formation of ternary [DOPE/DOPE:PEG(350)]-DNA-M²⁺ complexes that exhibit the inverted hexagonal phase, HIIc, in which the DNA strands fill the water space inside the cylindrical cavities of the DOPE/DOPE:PEG(350) hexagonal lattice. This result represents the first experimental evidence of a self-assembled formation of an inverted hexagonal complex structure in aqueous dispersions of DNA, metal cations and liposomes made of mixtures of pure and PEG-lipids. In addition, these experiments have shown that high concentrations of the PEG-lipid component destabilize the inverted hexagonal phase towards either a lamellar phase, in the absence of metal cations, or a novel inverted cubic phase (Q224 with space group Pn3m), in the presence of metal cations.

The second lecture given by F. Briki highlighted protein structure imaging in biological tissues using synchrotron X-ray microdiffraction. Mechanical stress effects will then be imaged across a mature hair shaft section showing keratin intermediate filaments molecular transformation from α -helices to β -sheets. Keratin intermediate filaments behaviour in the keratinisation zone during the differentiation process and the fibre formation will also be presented. She nicely explained how to follow the various keratinisation steps at molecular and supra-molecular levels. In a second example, she presented the results concerning internal structures of an amyloid deposit. Only by using a micron-sized beam to locate fibrous zones inside in vitro preparation as well as inside an amyloid laden tissue (kidney cut). The kidney cut was used to perform a first study of an amyloid deposit directly in human renal tissue sections, avoiding any possible extraction-induced artefact. Using X-ray microdiffraction, this revealed the presence of the typical cross- β - features from a frozen sample, similar to those observed for in vitro formed or extracted fibres. Moreover, mapping through the tissue section shows intrinsic orientation of the fibres

with a micrometer scale resolution, which interestingly is correlated to the glomerulus morphology.

P. Clegg as the third lecturer presented his results on dynamics of a colloid-stabilized cream using XPCS. The systems investigated, dodecane-in-water emulsions, have interfaces stabilized solely by colloidal particles (silica). They were observed soon after mixing: as the emulsion becomes compact, two regimes of ageing with a cross-over between them can be discerned. The young emulsion has faster dynamics associated with creaming in a crowded environment accompanied by local rearrangements. The dynamics slow down for the older emulsion although his studies show that motion is associated with large intermittent events. The relaxation rate, as seen from the intensity autocorrelation function, depends linearly on the wave vector at all times; however, the exponent associated with the line shape changes from 1.5 for young samples to less than 1 as the emulsion ages. The combination of ballistic-like dynamics, an exponent that drops below 1 and large intermittent fluctuations has not been reported before.

Finally, the session was closed by a very active and stimulating discussion of the partnership for SCM. The key message is, that the success of the implementation depends almost solely on how it is done. In detail, this means to bring support beforehand via memorandi of understanding, setting milestones and goals as well as - after the built-up - the facility of accessing the laboratories. Following the suggestions brought forward by T. Narayanan, additional instrumentation was demanded: environmental AFM, Raman spectroscopy, rheometer, fluidics, which ideally should be used online (which is already the case for Raman and fluidics) for N as well as X experiments. Concerning the staffing, a close collaboration with the user community in terms of longer-term stays of external visitors was strongly recommended, with benefits for both ESRF and users. On the one hand, placing external visitors at the center associated with the partnership allows for optimum use of sample preparation time and allows for introducing specific user knowledge of complementary key methods into the center. On the other hand, the access to combined N+X beamtime is facilitated by placing directly PhD-students there. A number of 15-20 visitors per year should be foreseen in the budget.

To conclude, the partnership for SCM was extremely well received. The external user groups present at the discussion immediately displayed their agreement of sending staff on a longer-term visitor basis to this center, manifesting their deep and close involvement in the ESRF'S future.

Soft Condensed Matter – parallel session

Organizers:

Theyengeri NARAYANAN (ESRF Group Leader)

Stephan ROTH (HASYLAB Hamburg, Germany & Users Organization) [stephan.roth@desy](mailto:stephan.roth@desy.de)

WEDNESDAY, 4 FEBRUARY 2009 14:00 - 16:00		
14:00 - 14:30	Report on the partnership for Soft Condensed Matter (PSCM)	Theyengeri Narayanan , ESRF, Grenoble, France
14:30 - 15:00	"Supramolecular structure and phase behavior of self-assembled liposome-DNA-metal complexes for gene transfer"	Oriano Francescangeli , Università Politecnica delle Marche, Ancona, Italy
15:00 - 15:30	"Proteins structure imaging in biological tissues using synchrotron X-ray microdiffraction"	Fatma Briki , University of Paris Sud, Paris, France
15:30 - 16:00	"Dynamics of a colloid-stabilized cream"	Paul Clegg , University of Edinburgh, Edinburgh, UK



Programme of the SCM parallel session

No parallel session was organized for the X-RAY ABSORPTION AND MAGNETIC SCATTERING group, as potential attendees were expected to participated at the workshop “Energy Dispersive X-ray Absorption Spectroscopy: Scientific Opportunities and Technical Challenges”