

## **ESRF Users' Meeting 2011: reports from Parallel Sessions**

**STRUCTURE OF MATERIALS**, held in the frame of the workshop "X-Rays and Neutrons in Energy-Related Material Science"

Organizers and chairs: Chiara Maurizio (ESRF Users Organization and University of Padova, Italy) and Andy Fitch (ESRF Group Leader)

The parallel session was attended by about 50 people. The topics of the talks were focused on the investigation of new complex materials promising for application in renewable energy and environment-compatible technologies.

The first two talks focussed on hydrogen storage materials, in particular investigating the structure and dynamics of complex hydrides (Andreas Borgschulte, Dübendorf, Switzerland) also using in-situ diffraction (Francesco Dolci European Commission, The Netherlands). Then the presentation of Susan Schorr (Free University Berlin, Germany) was on structural phase transitions in photovoltaic materials, with an in-situ high temperature synchrotron X-ray diffraction study. The last talk addressed another kind of environment-compatible materials, in particular high-strength steels for automotive applications, which was studied by high-energy x-ray diffraction.

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.

### **ELECTRONIC STRUCTURE & MAGNETISM**

Organisers and chairs: Carmela Aruta (Users Organisation & CNR-INFN, Napoli, Italy), Nick Brookes (ESRF Group Leader)

The parallel session on Electronic Structure & Magnetism was attended by about 40 people. Three invited talks were given by users that overviewed relevant scientific topics covering the activity of different ESRF beamlines in the field of the Electronic Structure and Magnetism. The first talk was given by Javier Garcia-Barriocanal on the remarkable study of the electronic, magnetic and orbital interface structures of Ti and Mn ions at  $\text{LaMnO}_3/\text{SrTiO}_3$  interfaces, mainly based on measurements performed at ID08. The second talk was given by Denis Andrault who showed the interesting results obtained on the Fe K-edge XANES spectroscopy at ID24 and the x-ray diffraction at ID27 to investigate the major phase relations between the different lower mantle minerals. The last invited talk was given by Fernando Bartolomé on the magnetism of noble metal nanoparticles investigated mainly by XMCD thanks to the very high magnetic field available at ID12. All the seminars were followed by vigorous debates.

Finally, a concluding talk was given by Nick Brookes on the group overview and the upgrade programme especially in view of the ESRF budgeted reduction provided for the period 2011-2013.

The session ended at 15:30 to allow interested users to join the workshop on multiferroics.

## MACROMOLECULAR CRYSTALLOGRAPHY

Organizers: Sean McSWEENEY (ESRF Group Leader), Gerlind SULZENBACHER (AFMB-CNRS, Marseille, France & Users Organization)

The Macromolecular Crystallography (MX) parallel session was held on 10 February 2011 from 14:00 until 18:00 at ILL Chadwick Amphitheatre and was attended by about 40 people. The first part of the session was devoted to information to the Users about the Upgrade programme of the Structural Biology beamlines, delivered by Christoph Mueller-Dieckmann, Petra Pernot, Matthew Bowler, all members of the ESRF Structural Biology Group.

It was brought to the attention of the Users the grouping of beamlines and facilities within the "Structure Village", that will include the new crystal screening MASSIF, the former ID14-3 BioSAXS station and the Cryobench. This arrangement, coupled with the setup of central facilities is foreseen to catalyze resource optimization.

Christoph Mueller-Dieckmann gave an overview of the future Structural Biology beamlines in the context of ESRF upgrade, including the adjustments brought by the recent budget reduction, that has led to postponing the building of one crystallography BL (Chartreuse Extension).

The future setup of the BioSAXS beamline was described by Petra Pernot, it will retain the main feature of ID14-3, with the introduction of novel optics leading to reduced divergence due to beam focusing onto the detector.

Matthew Bowler detailed the setup for the MASSIF facility, devoted to screening of crystals, also within microplates. Three fixed wavelength beamlines will be operative, two with 100  $\mu\text{m}$  and one with 10  $\mu\text{m}$  beam size, at a wavelength compatible with Se SAD phasing.

The Users requested a calendar on the ESRF website listing all the shut downs in order to organize the experimental calendar accordingly, (general ESRF shutdown from 12/2011 to 3/2012).

Information was requested by the Users as to whether the implementation of size exclusion chromatography is foreseen for the BioSAXS line.

As far as the MASSIF facility is concerned, it was brought to the general attention by Users and agreed by ESRF staff that issues on the scheduling of test time and on eventual crystal storage facilities will be posed. The ESRF staff announced that these issues will be dealt with while the facility becomes operative.

Sean McSweeney and Matthew Bowler communicated to the Users that a process is ongoing to develop novel sample holders in order to meet the necessity to increase substantially the number of samples to be shipped, screened and stored for screening and subsequent data collection. Users and ESRF Structural Biology staff expressed the intention to strive towards a common sample holder standard shared among different synchrotron sources, compatibly with the time frame of the MASSIF facility.

Talk by Users were as follows:

From the genome to structure and function: "Treasure hunting in the genome of the heterotrophic marine bacteria *Zobellia galactanivorans*" Mirjam Czjzek (Station Biologique de Roscoff, France), "Human mitochondrial termination factor mTERF wraps around target DNA through a left-handed helical tandem repeat" Maria Solà (Molecular Biology Institute of Barcelona-CSIC, Spain), "Structure of the bacteriophage T4 long tail fiber needle-shaped receptor-binding tip", Mark J. van Raaij (Departamento de Estructuras de Macromoleculas, CNB-CSIC, Madrid, Spain), "Transcriptional regulation by conditional co-operativity mediated

by allosteric coupling between two disordered domains”, Remy Loris (Free University of Brussels, Belgium), “Structural dynamics of neuroglobin: from ligand binding to signaling”, Beatrice Vallone (University of Rome "La Sapienza", Italy).

Finally, Gerlind Sulzenbacher introduced Beatrice Vallone as the new representative elected by the Users for the Structural Biology community, the Users and ESRF Staff thanked Gerlind Sulzenbacher for her contribution and dedication to the ESRF User Organization as representative of the SB Community and Chairperson of the Organization.

## **SURFACE AND INTERFACES**

Organizers and Chair: Christian Kumpf (Users Organization Committee / Research Center Jülich, Germany), Jörg Zegenhagen (ESRF)

The Surfaces and Interfaces (SI) parallel session benefitted significantly from the new, extended format which allowed to put together a program of nine talks, four given by users reporting their very successful experiments and 5 by internal staff members on inhouse projects and the status of different beamlines. The session was attended by up to 65 people. The meeting was overshadowed by rumors about closing one of the most important beamlines for SI science due to financial problems of the ESRF. Unfortunately these rumors have been confirmed shortly after: beamline ID32 shall be closed for user operation at the end of the year. This very unfortunate decision is being heavily discussed since then. The SI community is deeply concerned since worldwide unique possibilities in the field will be destroyed, most importantly the exclusive combination of x-ray standing waves (XSW) with hard x-ray photo electron spectroscopy (HAXPES) which was established only recently. The bright perspective which has grown from the combination of these techniques and carried manifold plans of a large number of user groups is dashed to pieces, just like those of the growing community working with diffraction techniques on buried (solid-liquid) interfaces, e.g., in the field of electrochemistry. At present the situation is unresolved and, even though some possible solutions are discussed, there are no specific perspectives foreseeable.

However, during the SI parallel session excellent science dominated the atmosphere of the meeting rather than political jeopardies. The internal contributions were given by Thomas Cornelius (ID01), Jörg Zegenhagen (ID32), Roberto Felici (ID03), Veijo Honkimäki (ID15) and Pilar Ferrer (BM25). Highlights were investigations of InAs nanowires using the ptychography technique on the new ID01 diffractometer, the first experiments on the new HAXPES setup at ID32, which reaches 50meV resolution at 8keV kinetic photon electron energy and enables the XSW-imaging technique for a wide range of materials and systems, as well as the possibilities realized by the new 6K-cryostate and the heavy load (200kg) diffractometer at ID03. Furthermore we heard about experiments performed at the high energy beamline ID15 on liquid-solid surfaces (Pb-Si, In-Si), and on diffraction experiments on Isoleucine amino acids on Cu surfaces using the new electrochemistry cell at BM25.

The first user-talk was given by Herbert Over (Universität Giessen, GER) about the CO catalysis using RuO<sub>2</sub> surfaces. He put special emphasis on the correlations between the microscopic properties at the surface at an atomistic level and the activity of the catalytic process, and herewith explained the poisoning effect of the surface. In the second user talk Robert Feidenhans'l (Københavns Universitet, DEN), former head of the ESRF council and hence mostly seen on site for political reasons within the last years, reported on fascinating pump-probe experiments: Excited by a pulsed laser beam 50 nm thick GaAs nanowires start

“dancing”: Their crystallographic lattice “breathes”, i.e., their radius increases by up to 12 pm which can be resolved by time resolved x-ray scattering.

After the coffee break two more users talks were given: Carsten Busse (Universität zu Köln, GER) reported interesting details of the graphene synthesis on Ir(111): In a careful NIXSW study the buckling of the graphene sheet was measured quantitatively, and consequently its use as a template for ordered growth of Ir islands was investigated. Finally Ronan Guillamet (Alcatel-Thales III-V Lab, FRA) reported on high resolution X-ray micro-diffraction experiments using the zone-plate setup of ID01 for the investigation of metal organic chemical vapor phase epitaxy (MOVPE) systems on pre-patterned AlGaAs heterostructures. During this session we have heard many very interesting reports on fascinating investigations using surface / interface diffraction as well as spectroscopic investigations. The attendance of the users was as strong as usually in the last years. It is to be hoped that the very active and successful SI user community will not disperse due to the consequences of unfortunate political and financial boundary conditions, but that the responsible boards at ESRF succeed in maintaining the excellent conditions which have been established during the last years for the fields of SI science.

## **SOFT MATTER STRUCTURES**

Organisers and Chairs: Olivier Diat (User Organisation & ICSM, Bagnols s/Céze, France), Theyencheri Narayanan (ESRF Group Leader)

30 scientists were attended to this session with the presence of S. Perez, ESRF director of research that we appreciate for his answers to some questions. Before the 4 selected talks from users, the status and upgrades of the main ESRF beamlines involved in soft matter, i.e. ID02, ID10 and ID13 were presented by the beamline scientists respectively.

Concerning ID02, M. Sztucki has explained the new version of the upgrade (part of the UPBI9a) with a large effort towards very small angle of detection via a 2D detector that can be moved at 30m from the sample position. The possible use of a four bounce crystal collimator as well as lenses can provide a focalised beam at the beamstop position. Highly stable focusing optics based on a large toroidal mirror with meridional RMS slope error less than  $0.25 \text{ mrad}^2$  plus a high resolution detector with spatial resolution better than  $30 \text{ \AA}$  (FWHM) and single photon sensitivity (e.g. FReLoN based on large EEV sensor) would provide a significant improvement in TRUSAXS experiments.

O. Konovalov and Y. Chushkin have shown the ID10 BI upgrade which will provide a important gain in flux (10 x more), in energy range (7-30keV instead of 8-22keV) in term of focusing from *sub mm* to *sub  $\mu\text{m}$*  with an anomalous option, all of this optimization for ID10B for reflectivity measurements and a gain in the brilliance and coherence with a wavefront distortion minimization for ID10A . The main black point is the sharing of the beamtime which mainly prevent an access of the id10b hutch during ID10A. This will required from the ID10 staff a very efficient beamtime allocation and organization. A calendar of the upgrade was presented.

Then, the first invited talk should have been presented by Barbara RUZICKA from SOFT INFM-CNR and Dipartimento di Fisica, Sapienza Università di Roma who did not come for health reasons and so Dr Narayanan T. has presented the slides that were sent by Dr Ruzicka. The subject concerned “Competing Interactions in Arrested States of Colloidal Clays” and an explanation of a new gel state. Using experiments, theory and simulations, he

shows that the arrested state observed in colloidal clay at high concentrations is stabilized by screened Coulomb repulsion (Wigner glass). Dilution experiments allow us to distinguish this disconnected state, which melts upon addition of water, from a low-concentration gel state, which does not melt and is very stable (perhaps a new state). Theoretical modelling and simulations at high concentrations reproduce the measured small angle x-ray scattering static structure factors and confirm the long-range electrostatic nature of the arrested structure. These findings were attributed to the different time scales controlling the competing attractive and repulsive interactions.

The second invited talk was given by Thomas BLOCHOWICZ from the Technische Universität Darmstadt on the Concentration fluctuations in a binary glass former. This work was carried out using x-ray photon correlation spectroscopy to characterize the structure and dynamics of concentration fluctuations in the binary glass former methyl-tetrahydrofuran and oligomeric methyl metacrylate. Indeed, although the system was macroscopically well miscible and optically clear in the full temperature range, calorimetric and dielectric measurements reveal two distinct glass transition temperatures. The relaxation of long range concentration fluctuations turns out to be diffusive and exponential only well above the upper glass transition temperature. Moreover, the analysis of time-resolved correlation functions reveals that the relaxation of concentration fluctuations around  $T_g$  involves pronounced dynamic heterogeneities.

The third talk was given by Dr F. Rossetti who accepted to replace Pr. Tanaka from Prof. M. TANAKA (Institut für Physikalische Chemie, Universität Heidelberg) and to present her work on the Influence of Oligo- and polysaccharides on structures and mechanics of membranes using specular and off-specular scattering and fluorescence methods. This talk was divided in two parts, the first one concerning models of Cell-ECM Contact with Polymer-Supported Membranes. Interaction forces were evaluated through the distance between supported membrane and the substrate. The second part was dealing with mechanism of bacterial resistance against CAPs in the presence of calcium cations and the collapse effect of sugar type amphiphile.

Finally, a nice work on stresses in wood was presented by Dr B. Clair from University of Montpellier. This is a study performed on ID13, using a microbeam for answer to the questions, does cellulose support the tension? And when and how is cellulose put under tensile stress? It was a study with comparison between normal wood and tension wood of young growing poplar artificially tilted at the ESRF. B. Clair demonstrates that X-ray micro-diffraction allowed the exploration of changes in cellulose ultrastructure along cell wall maturation, that variations in intensity were consistent with the deposition of crystalline cellulose during the progressive thickening of the cell walls and the changes in cellulose organisation with apparition of G-layer.

These four talks were quite well representative of the possibility given by the three beamlines to provide nice and new data over a wide class of examples with soft structures. During the discussion slots, radiation damage were discussed as well as the coupling of different analysis like the structure and Raman spectroscopy already developed on id13. The PSCM with possible proposal and collaboration were also discussed!

**X-RAY IMAGING**, held in the frame of the workshop “X-Rays and Neutrons in Energy-Related Material Science”

Organizer: Josè Baruchel (ESRF Group Leader)

The programme of the workshop session dedicated to x-ray imaging took place with the following talks:

N. Mangelinck-Noel (IM2NP Marseille, France): “X-ray Synchrotron imaging characterisation of the solidification of materials devoted to energy saving issue. Photovoltaic silicon and metallic materials for transport applications”.

H. Palancher (CEA Cadarache, France) “Diffraction and 3D imaging of low-enriched nuclear fuels”.

C. Linsmeier (MPI München, Germany): “SR tomography on porous tungsten for nuclear fusion reactors”.

L. Luquot (University of Montpellier, France): “Characterization of structural rock properties changes due to CO<sub>2</sub> rich-brine injection by X-ray microtomography”.

### **DYNAMICS & EXTREME CONDITIONS**

Organisers and Chairs: Chrystèle Sanloup (User Organisation & University Paris-6, France), Tullio Scopigno (User Organisation & University Roma – La Sapienza, Italy), Michael Krisch (ESRF Group Leader)

The session gathered over 40 people, started with an introduction by the DEC group leader and was followed by five 20 minutes talk by invited speakers covering a large spectra of the activities carried on the DEC beamlines.

Michael Krisch presented us the latest improvements and innovations on the beamlines, thus widening experimental opportunities. Those include large volume press experiments in the near future as the press is now being commissioned at the ESRF, installation of a CO<sub>2</sub>-laser heating system and of a He-cooled cryostat for diamond-anvil cell experiments on ID27, a high resolution MAR550 image plate on ID09, enhancement of on-line diffraction capabilities on ID28. The targets for future ID20 beamline (replacing the existing ID16) were presented.

Talks by invited users covered the following topics : inelastic X-ray scattering on high-pressure fluids (F. Gorelli, Univ. Florence, Italy), phase diagrams of Lithium and Sodium at extreme pressure (C. Guillaume, Univ. Edinburgh, UK), melting curve of peridotite up to 140 GPa (G. Fiquet, Univ. Paris 06, France), supported antiferromagnetism in FeO nanolayers by in-situ nuclear resonant scattering (S. Couet, KU Leuven, Belgium), K-edge magnetic circular dichroism in 1s2p resonant inelastic X-ray scattering (M. Sikora, Univ. Krakow, Poland). Each of the talks was followed by questions and comments, showing crossed interests over the DEC users' communities.