

## ESRF USERS' MEETING 2012: REPORTS FROM PARALLEL SESSIONS

### STRUCTURAL BIOLOGY PARALLEL SESSION

**Organizers and Chairs:** Beatrice VALLONE (Users' Organization and Dept. of Biological Sciences, University of Rome "La Sapienza", Rome, Italy), Sean McSWEENEY (ESRF Group Leader).

The Structural Biology parallel session was held on 7 February 2012 from 14:00 until 18:30 in the seminar room of the Carl-Ivar Bränden Building and was attended by about 50 people.

The first part of the session was devoted to the new beamlines setup and to the design and integration of software for the design of experiments in the context of the upgraded MX facility, delivered by Christoph Mueller-Dieckmann and Alexander Popov. Both talks raised considerable attention from the Users and it was decided that the final part of the session would be extended and devoted to questions on these matters.

Talk by Users were as follows:

-“The alternate assembly of retroviral integrases?” Patrice Gouet, Institut de Biologie et de Chimie des Protéines (CNRS/Université Lyon- France),

-“Structural Basis of Tumor Suppressor in Lung Cancer 1 (TSLC1) Binding to Differentially Expressed in Adenocarcinoma of the Lung (DAL-1/4.1B)”, Martin Hällberg, Department of Cell and Molecular Biology, Karolinska Institutet Stockholm, Sweden.

-“The structure and mechanism of two proteins involved in DNA repair: UvrA and RNase H2”, Marcin Nowotny, International Institute of Molecular and Cell Biology, Warsaw, Poland.

-“Structural and spectroscopic observation of an enzyme at work” Andrea Mattevi, Department of Biology and Biotechnology, University of Pavia, Pavia, Italy.

From 18:00 until 18:45 Sean McSweeney led and moderated a lively session with the users, who posed several questions dealing on the strategy to adopt with the respect of the increased throughput that will be achieved by the MASSIF setup for MX beamlines.

The agreement on the advantages of increasing substantially the number of samples shipped and on the strategic value of crystal screening and characterization, with the implementation of software tools to design the best data collection strategy, was general.

Much interest was expressed on the design of new sample holders and the Users expressed the wish that interaction with the users' community would take place during this process and, more in general, in the organization of sample flow in the new setup.

The Users requested that a chart that would clarify the time evolution of beamlines shutdown and opening was made available on the ESRF web site, in order to facilitate programming of experimental sessions for the users. It was understood that such a chart could not be precise, given the unpredictability of some of the upgrade operations.

There was a request on information about project evaluation and priority for beam time allocation to structural biology on non MX beamlines.

At the end of the Plenary Session of the 2012 User Meeting (8 February 2012, 11:30-12:00), an open discussion was held, with members of the Users Organization addressing questions for the different communities to the ESRF Directorial Panel

### X-RAY IMAGING PARALLEL SESSION

**Organizers and Chairs:** Eric Maire (Users' Organization and CNRS-INSA, Lyon, France), José BARUCHEL (ESRF Group Leader).

The session gathered about 30 people, which is consistent with the average number seen every year. The session started with a short informative talk given by Jose Baruchel. He explained to the gathered users the progress of the upgrade program (especially NINA) and he gave us an update about the clinical trial which are about to be authorized on ID17 by the French administration. The first invited speaker was Isabelle Rouget who showed us how imaging can be used to analyze the content of old fossils (published in science). The second speaker, Koen Janssens showed us how X ray fluorescence tells a lot about the hidden content of Van Gogh's and Rembrandt's paintings (high coverage in the international press). The third speaker, Emmanuelle Gouillart, uses fast tomography to study the fabrication of Silica glass from melting sand. The fourth speaker L. Siller is studying the effect of silver

nano-particles on the development of cells. After the coffee break, J Segura-Ruiz showed us how fluorescence with the very robust new focussing device (to be used on NINA) is now operational to produce fine X-ray spots. Finally, Alexander rack presented the imaging group project for the establishment of a phase contrast, ultrafast and large field of view imaging station at the ESRF during the phase II of the upgrade program.

#### **DYNAMICS & EXTREME CONDITIONS PARALLEL SESSION**

**Organizers and Chairs:** Chrystèle Sanloup (User Organisation & University of Edinburgh, UK), Michael Krisch (ESRF Group Leader).

The session gathered over 30 people, started with an introduction by the DEC group leader, was followed by six 20 minutes talk by invited speakers covering a large spectra of the activities carried on the DEC beamlines, and was concluded by a discussion of the phase 2 of the ESRF upgrade. M. Krisch presented us proposed beamline developments for phase 2, including the extension of ID27 with the construction of a 3rd hutch potentially dedicated to laser shock experiments, nanotomography in the Paris-Edinburgh cell, or nano XRD and X-ray fluorescence techniques for diamond-anvil cells (DAC) experiments.

Wishes and concerns from users to the management for phase II are reported in a dedicated document.

#### **STRUCTURE OF MATERIALS PARALLEL SESSION**

**Organizers and Chairs:** Chiara Maurizio (User Organisation & University of Padova, Italy), Andrew Fitch (ESRF Group Leader).

The parallel session gathered about 35-40 people and started with report from ID11, ID15 and ID31 beamline scientists (G. Vaughan, V. Honkimaki and A. Fitch, respectively) about last developments on the beamlines. Then four invited talks followed. The first was by Simon Billinge, of Columbia University and BNLS, on the use of atomic pair distribution function with high energy x-rays. The two following talks dealt with a combination of x-ray based technique to fully characterize materials, in particular, Andrew King (ESRF) talked on the combined use of X-ray imaging and diffraction techniques for the investigation of deformation and damage mechanisms at the grain scale, while Simon D.M. Jacques (University of Manchester, UK) reported on the development of hyper-spectral x-ray imaging for chemical and material science. The last talk, given by Boaz Pokroy (Technion, Israel Institute of Technology), was on the structure and microstructure of biogenic and biomimetic nanocomposites, showing how organic/inorganic interfaces can control the crystalline structure of biominerals. A lively discussion followed each talk, in particular on the potential and limitation of the employed techniques.

#### **SOFT MATTER STRUCTURE PARALLEL SESSION**

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

35 scientists were attended to this parallel session. Before the 5 selected talks from users, a short status and upgrades foreseen were given for ID02 (see design report UPBL9a on the following web address <http://www.esrf.eu/AboutUs/Upgrade/future-beamline-portfolio>) with some questions relative to microbeam and micro-radian divergence possible with the improvement of the beam emittance and for ID10 (see upgrade programme – phase 1, conceptual design report) with improved management of beam use (50/50 %) between both stations of the beamline. Then D. Pontoni has explained and presented the status of the PSCM with the final agreement process that will allow starting some common projects between ESRF and ILL with SC users. This was received as good news by the SC community and information can be found via this web address at that time (<http://www.ill.eu/?id=156>) in waiting an updated and new web site.

Then, the first invited talk should have been presented by Dr. S. Santucci from Trieste and who has spent a lot of her time on ID13. The subject concerned “Optical sample manipulations for

synchrotron radiation probing of biological and soft matter objects in aqueous environments". She presents how Optical tweezers (OT), based on the trapping capabilities of focused laser beams, have found numerous applications in different fields from physics to life sciences. She shows us how on ID13 they have realized and commissioned an OT setup adapted to the synchrotron environment at the ESRF. The performance of the setup in the beamline environment has been tested by WAXS and SAXS scattering experiments on single optically trapped starch granules, using a synchrotron radiation microfocussed beam at the ID13 beamline. She has explored the feasibility of microdiffraction on optically trapped protein crystals of 35 microns diameter. The effect of radiation damage at room temperature has been investigated by repeated raster-scanning up to the complete loss of the structural order. And finally, she demonstrate that in the near future, the combination of optical forces, synchrotron and laboratory probes may initiate the development of new research fields for fragile, biological and soft matter objects.

The second invited talk was given by Prof. John Miao from Department of Physics & Astronomy and California NanoSystems Institute University of California, Los Angeles, USA on "Coherent Diffraction Imaging of Biological Materials. Coherent diffraction imaging (CDI) is a lensless imaging technique in which the diffraction pattern of a noncrystalline specimen or a nanocrystal was first measured and then directly phased to obtain a high-resolution image. He shows us how the well-known phase problem was solved by combining the oversampling method with iterative algorithms. In this talk, he first presents the application of CDI to the imaging of mineral crystals inside intramuscular fish bone at the nanometer scale resolution. Mineral crystals in collagen fibrils at different stages of mineralization were identified and a dynamic model for the nucleation and growth of mineral crystals in a collagen matrix was proposed. The he reports on their recent experimental results on the imaging of frozen-hydrated biological cells, which were obtained from ID10 at the ESRF.

The third talk was given by Dr Marianne Imperior-Clerc from LPS at Orsay on "Self-assembly in solution of silica-based hybrid materials: Contribution of *in-situ* SAXS studies". Surfactant templated mesoporous silica powders have been extensively studied in the past 15 years by exploiting the self-assembly properties of amphiphilic molecules in aqueous medium. The large success of these systems mainly relies on the apparent simplicity that enables to design porous networks with different architectures and pore sizes. However, a better control of the final nanostructure (2D-hexagonal, 3D-cubic) requires a deeper understanding of the interaction mechanisms between the templating agents and the network-forming species. This contribution presented their latest experiments using *in-situ* Small Angle X-ray Scattering (SAXS on Soleil and BM2) performed with highly brilliant synchrotron radiation to follow the formation mechanism of templated silica powders in the presence of different types of surfactants (non-ionic triblock copolymers, non-ionic fluorinated surfactants, cationic surfactants with different polar head groups) and silica precursors. This type of experiments gives access to a lot of information about the cooperative self-assembly of these systems and the nanostructure formation. She emphasizes how the inorganic precursor affects the micelles in solution (prior to, during and after hydrolysis of the precursor) and how the micelles shape is linked to the final nanostructure of the hybrid material.

After a coffee break, the fourth talk was dedicated to "Muscle contraction probed using high resolution and TR-SAXS". Combining time-resolved SAXS and fast mechanics in single cells dissected from the skeletal muscle of the frog, we were able to determine the load dependence of the amplitude and the speed of the working stroke in the myosin motor, the mechanism of muscle braking in response to stretch and the conformational change in the myosin motors during muscle activation and the development of isometric force (Reconditi et al., *PNAS USA*, 108:7236-7240, 2011). Recently they extended this approach to mammalian muscle, using either demembrated fibres from rabbit psoas, in which the biochemical milieu can be controlled, or the whole mouse muscle, in which an adequate signal to noise is achieved for the weaker X-ray reflections that carry fundamental information about the structural changes in the filaments. This has enabled a new programme of research, linking the structural and mechanical changes in the myosin motor to the steps of the ATP hydrolysis cycle that fuels contraction. In future the combination of SAXS with transgenics and transfection of mouse muscles will allow the investigation in intact muscle cells of the structural and functional consequences of specific molecular changes relevant to human diseases.

Finally, a nice work on "Gas Transport through Smut Formed During AC Electrograining by Synchronized SAXS and electrochemical experiments." Was presented and show us how large is the domain of soft matter and close also to very different application (corrosion in that case). Dr J.A Hammons from the group of Electrochemical and Surface Engineering at the Vrije Brussel university,

explains us how SAXS was used to characterize the particles and surrounding phases inside a surface gel that inherently forms during the AC electrograining process. By synchronizing the electrochemistry with the SAXS data, his team was able to obtain the potential response during electrograining and the rate of gas release (from the gel) after electrograining. Analysis of these results shows that gas transport, through this gel, and dried smut morphology both change when different additives are used. Two general gas transport mechanisms are proposed, which explain differences in the experimental results. Finally, he shows that differences in gas transport and final surface morphology indicate that gas transport through the surface gel may be an important factor in the AC electrograining process.

These five talks were quite well representative of the possibility given by the beamlines to provide nice and new data over a wide class of examples with soft structures.

During the discussion slots, radiation damage were discussed especially when very tiny beam is used. Also the possibility to exploit more the coherent part of the beam was discussed and will be perhaps the subject of a next UM satellite workshop.

### **ELECTRONIC STRUCTURE AND MAGNETISM PARALLEL SESSION**

The parallel session of Electronic Structure and Magnetism was integrated with the workshop "Magnetic Materials under Extreme Conditions".

#### **Workshop Organizers:**

C. Aruta, ESRF Users' Organisation & CNR-SPIN, Napoli, Italy, J. P. Attfield, ESRF SAC member & University of Edinburgh, UK, N. Brookes, ESRF group leader  
C. Detlefs, ESRF, A. Kaprolat, ESRF, S. Pascarelli, ESRF, A. Rogalev, ESRF, F. Wilhelm, ESRF

The aim of the workshop was to bring together the communities of scientists investigating magnetic materials ranging from low dimensional systems up to macrosized samples under extreme conditions. The workshop covered a broad spectrum of topics embracing fundamental and theoretical aspects of phenomena occurring under high magnetic field, electric field, high pressure and strong optical fields in combination with low or high temperature.

The workshop was opened by Harald Reichert, Director of Research at the ESRF, to illustrate the upgrade program and in particular the phase two of ESRF.

Most of the speakers were synchrotron users (either ESRF, Soleil, SLS,...). The invited speakers were specialists of a given field to present the importance and necessity to measure magnetic materials under extreme conditions. All of them showed that a combination of extreme conditions (low T and high H, low T and High P, etc..) are necessary to deeper understand the magnetic properties of magnetic materials.

At the parallel session on Tuesday, February 7, three invited and two contributing talks were given:

C. Binek (invited) "Isothermal electric control of exchange bias near room temperature",

D. McMorrow (invited) "Quantum phase transitions in low-dimensional model magnets"

A. Kimel (invited) "Generation of femtosecond pulses of effective magnetic field for ultrafast control of magnetism"

R. Torchio (contributing) "Pressure induced magnetic and structural transitions in the 3d metals and their alloys by means of K-edge XMCD"

J. E. Hamann-Borrero (contributing) "Magnetic structure of magneto-electric  $\text{NdFe}_3(\text{BO}_3)_4$  under applied magnetic and electric fields.

The number of participant was 76 in total and the scientific debate was very lively during all the sessions of the workshop.

### **SURFACES AND INTERFACES SCIENCE PARALLEL SESSION**

**Organizers and Chairs:** Christian Kumpf (Users Organization Committee & Research Center Jülich, Germany), Jörg Zegenhagen (ESRF)

Following last year's tradition the Surfaces and Interfaces (SI) parallel session was put together from two types of contributions. In four presentations ESRF staff members reported the status of their beamlines and some scientific inhouse projects. Three talks were given by ESRF users on their recent

prominent experiments. The session, which for the first time was held in the ESRF auditorium, was attended by about 80 participants.

Recent decisions of the ESRF steering committees and the management, and their consequences for the SI community were a dominant subject in the discussions. Beamline ID32, one of the most important beamlines for SI science that had offered worldwide unique possibilities in the field of x-ray standing waves (XSW) and hard x-ray photo electron spectroscopy (HAXPES), has been closed for user operation at the end of 2011. A dedicated XSW and HAXPES workshop, which was held as a satellite meeting of the 2012 users meeting, was aimed at shaping plans for a new HAXPES/XSW beamline within the frame of the phase two of the ESRF upgrade. The SI parallel session was officially included in the workshop program, and turned out to be – just like the workshop itself – a great success in terms of number of participants and quality of work presented. But even though the community was excited about the “upgrade-phase-II” idea, this solution will take quite some time. Thus, for several years there will be no beamline available for the XSW/HAXPES community, with the consequence that it may be lost for the ESRF. Even more so, due to missing alternatives, – the community might break apart and lots of knowledge and experience will be lost. This can only be avoided by offering an immediate interim solution until an electron spectroscopy XSW beamline reopens at the ESRF. Hence, during the users meeting and the satellite workshop the SI community has made a strong case for a fast if not immediate reopening of a successor beamline of ID32, in particular if the financial situation of the ESRF improves allowing the original number of ESRF-operated beamlines to be re-established. A HAXPES/XSW beamline within the frame of upgrade phase II is strongly appreciated, but a strategy needs to be developed to allow the access to XSW/HAXPES facilities for the community earlier than this.

Beside this more political aspect, which was heavily discussed during the meeting, scientific results were standing in the focus of the parallel session. Internal contributions were given by Dina Carbone (ID01), Olivier Balmes (ID03), Pilar Ferrer (BM25) and Valentina Cantelli (BM32). Highlights from the insertion device beamlines were anomalous coherent x-ray diffraction results on Ag-Au core-shell nanowires, crystal truncation rod measurements of CO adsorbed on Pt(779) surfaces (both ID03), nano-beam diffraction experiments on Si/SiGe nanostructures inside a field effect transistor, and first experiments on x-ray scanning diffraction microscopy, Bragg holography and Bragg ptychography (at ID01). Furthermore we heard reports on the characterization of BiFeO<sub>3</sub> single crystals using a combination of HAXPES and XRD (BM25), as well as Si nanowires on Si, and InAs and InP on GaAs at BM32.

Three longer talks were given by ESRF users. Edwin Lundgren (University of Lund, Sweden) reported highly interesting in-situ studies of model catalysts for CO and CH<sub>4</sub> oxidation. He presented investigations of the surface structures of Pd surfaces, the reconstructions of which were monitored in situ, and pointed out the specific need of combining the HAXPES technique with ambient pressure conditions for future investigations of catalysts. This would be able to push the field forward and reduce the gap between catalysts operating under real conditions and laboratory experiments.

In the second presentation Moritz Sokolowski (University of Bonn, Germany), gave an excellent overview on the XSW activities of his own and other groups at beamline ID32 within the last decade. He put organic materials adsorbed on coinage-metal surfaces in the focus of his presentation and emphasized two important aspects: At first, the large importance which NIXSW results have gained as benchmark experiments for the understanding of metal-organic contacts, in particular regarding theoretical studies with ab-initio methods, and secondly, the enormous progress which has been made at beamline ID32 within the last two years when the new HAXPES analyzer became available. The latter aspect emphasizes that ID32 has been closed at the most unbearable time, just at the height of a long-years user-driven development.

After the coffee break there was one more user's talk given by Grzegorz Kozłowski (IHP Microelectronics Frankfurt/Oder, Germany). Within the general goal of achieving better optoelectronic properties by implementing Ge in well established Si process technology he reported several new growth techniques for Ge nanostructures on SiO layers on Si, Ge/Si nanopillars and Ge

crystals on Si pillars. He highlighted high precision measurements of stress and strain in these nanostructures and the importance of surface sensitive x-ray scattering techniques like x-ray reflectivity and reciprocal space mapping in this context.

Very interesting contributions from all speakers made the SI parallel session a successful event. Scientific and technical aspects were lively discussed by the participants, as well as the more political aspects around the situation regarding ID32. Like in last year's report on the SI parallel session we would like to conclude by expressing our hope 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."