



Swiss-Norwegian Workshop on Synchrotron and FEL-Based Research

Scandic Oslo Airport
Gardemoen, Norway
6th–7th June 2017



Organizing committee:

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Vladimir Dmitriev

SNX, SNBL, PSI
and the Swiss
and Norwegian
user community



10:30-11:25	Registration	
11:30-12:30	I Part: Background for the workshop	
11:30-12:00	Helmer Fjellvåg	
12:00-12:30	Rafael Abela	<i>"Developments at PSI"</i>
12:30-13:15	Lunch	
13:15-16:10	General Session: ongoing developments at SNX, ESRF, SNBL, and PSI and their impact on each other	
13:15-13:45	Vladimir Dmitriev	<i>"SNBL Today and Tomorrow"</i>
13:45-14:15	Meitian Wang	<i>"Development of Macromolecular Crystallography Beamlines at the SLS"</i>
14:15-14:45	Nicola Casati	<i>"The Material Science beamline at the SLS and SLS-2"</i>
	coffee break	
15:00-15:20	Ragnvald Mathiesen	<i>"Hard X-ray Transmission Microscopy"</i>
15:20-15:40	Reidar Lund	<i>"From Non-equilibrium to Equilibrium: Kinetic Processes in Self-assembled Systems seen by Time-resolved Small-angle Scattering"</i>
15:20-15:50	Erik Wahlström	<i>"A possible roadmap for establishing of Norwegian user organization promoting and facilitating the Norwegian use of synchrotrons, free-electron lasers and neutron sources"</i>
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	coffee break	
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16:30-16:50	David Wragg	<i>"Operando studies of Battery Materials at the ESRF"</i>
16:50-17:10	Grigory Smolentsev	<i>"Development of time-resolved X-ray absorption spectroscopy at SuperXAS beamline to study photocatalytic reactions"</i>
17:10-17:30	Magnus H. Sorby	<i>"Structure and properties of energy materials at SNBL and Neutron"</i>
	coffee break	
17:45-18:05	Radovan Cerny	<i>"Metal hydrides for energy storage"</i>
18:05-18:25	ZÜTTEL Andreas	<i>"X-Rays for Energy Materials"</i>
18:25-18:45	Diana Lucia Quintero Castro	<i>"Plans and Challenges in the Investigation of the Crystallographic and Magnetic Structures of Itinerant and Strongly Correlated Electron Systems"</i>
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20:00	Dinner	

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09:20-09:40	Paula Abdala	"Understanding bimetallic catalysts under working conditions by in situ combined XAS-XRD-MS"
09:40-10:00	Anja O. Sjøstad	"Nanomaterials for Catalysis"
10:00-10:20	Mark Newton	"XAFS, XRD and PDF at SNBL: application to catalytic materials in operando at a unique resource"
10:20-10:40	Discussions	
coffee break		
11:00-11:20	Anders Bank Blichfeld	"In-situ toolbox for studying nucleation and growth in oxide thin films and hierarchical structures prepared from aqueous solutions"
11:20-11:40	Andreas Voegelin	"Using XAS to elucidate the biogeochemistry of trace elements in environmental systems"
11:40-12:00	Pascal Dietzel	"Investigation of the host-guest chemistry in coordination polymers using in-situ and operando experiments"
12:00-12:20	Discussions	
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13:35-13:55	Ana Diaz	"High-resolution 3D imaging at the cSAXS beamline using X-ray ptychographic tomography"
13:55-14:15	Hans-Petter Hersleth	"Combining Protein X-ray Crystallography and in situ Single-Crystal UV-Vis and Raman Spectroscopy to Grasp the True Structure of Haem- and Flavoproteins"
14:15-14:35	Ioana Bradeanu	Skin penetration study of drug carriers using soft X-Ray spectromicroscopy
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Developments at PSI

Rafael Abela

Paul Scherre Institute, 5232 Villigen PSI, Switzerland

Abstract

The status and development plans for the large scale facilities at PSI - SwissFEL and SLS – will be presented. The talk will focus on new research opportunities and capabilities, challenges to be addressed as well as collaboration schemes.

SNBL Today and Tomorrow

V.Dmitriev, D.Chernyshov, V.Dyadkin, H.Emerich, W. Van Beek

The Swiss-Norwegian beamlines at ESRF, Grenoble Cedex 9, France

Abstract

We will present the status of the Swiss-Norwegian Beamlines at ESRF and development plans for it in view of the upgrade to new ESRF Extra Brilliant Source. The talk will analyse the opportunities offered by the existing setups at BM01 and BM31 and suggest their developments. Budgeting restrictions make us being realistic.

Development of Macromolecular Crystallography Beamlines at the SLS

Meitian Wang

Swiss Light Source at Paul Scherrer Institut, CH-5232 Villigen PSI

Abstract

Three Macromolecular Crystallography (MX) beamlines at the Swiss Light Source, collectively delivered 5,000 PDB entries to date, are among the most productive MX beamline worldwide. A science-driven and instrumentation-enabled approach is used to address a broad range of challenging projects in structural biology. The Recent development of micro-focusing X-ray optics, multi-axis goniometers (PRIGO/SmarGon), single photon counting detectors (PILATUS/EIGER) and their applications in serial synchrotron crystallography and native-SAD phasing will be presented. New opportunities in SLS-2 and SwissFEL will be discussed as well.

The Material Science beamline at the SLS and SLS-2

Nicola Pietro Maria Casati

Beamline scientist – MS beamline, Swiss Light Source at Paul Scherrer Institut, WLG/229CH-5232 Villigen PSI

Abstract

The Material Science beamline traditionally serves academic and industrial users for powder diffraction experiments, especially in time-resolved and high-statistics, high-resolution measurements. Novel capabilities are planned and will be discussed, in view of a novel MythenIII detector design and of the planned upgrade to a diffraction limited SLS-2 source.

Hard X-ray Transmission Microscopy

Ragnvald Mathiesen^a, Ken V. Falch^a, Daniele Casari^a, Henning Friis Poulsen^b, Hugh Simons^b, Carsten Detlefs^c, Marco Di Michiel^c, Irina Snigireva^c, Veijo Honkimaki^c, Anatoly Snigirev^d

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^d *Immanuel Kant Baltic Fed. Univ, Russia*

Abstract

A hard X-ray transmission microscope based on compound refractive lenses is being built at the ESRF. The microscope can operate both in dark field and bright field, and is designed to allow for an hierarchical multi-scale approach to the studies of materials and materials processes. The instrument is designed such that also broad band radiation may be employed without too severe chromatic effects, which allows for a unique combination of high resolution and fast image acquisition.

From Non-equilibrium to Equilibrium: Kinetic Processes in Self-assembled Systems seen by Time-resolved Small-angle Scattering

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The kinetic pathways associated with the formation, transformation and equilibration of self-assembled nanostructures are not fully understood. Time-resolved small-angle X-ray/neutron scattering (TR-SAXS/SANS) is a powerful technique¹ that allows kinetic processes such as nucleation processes^{2,3} and morphological transitions^{4,5} to be followed with structural resolution over time scales starting from milliseconds. Neutrons offer the additional advantage of facile contrast variation through H/D substitution schemes, which also allow equilibrium processes such as molecular exchange and diffusion to be studied^{1,6,7}.

Here we will highlight the current capabilities of TR-SAS and show examples from the kinetics of micellar systems. Moreover, we will address how the understanding of kinetic pathways can be used to control the nanostructure.

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7. S. H. Choi, et al. *Phys Rev Lett.* 2010, 104, 47802.

A possible roadmap for establishing of Norwegian user organization promoting and facilitating the Norwegian use of synchrotrons, free-electron lasers and neutron sources

Erik Wahlström

Department of Physics, Norwegian University of Science and Technology (NTNU)

Abstract

A short overview of the needs to establishing a general user organization promoting the use synchrotrons, free-electron lasers and neutron sources will be made. The possible aim and roles will be discussed, a possible route for establishing it will be outlined with the aim to initiate such an organization during 2017.

Operando studies of Battery Materials at the ESRF

David Wragg, University of Oslo

If we could store the energy generated from renewable sources more effectively our dependence on fossil fuels could be significantly reduced. This requires us to develop better materials. *Operando* studies are an essential tool in the search, giving us understanding of how structure controls properties under real working conditions. In this talk I will describe several works on *operando* characterisation of electrode materials for sodium batteries. The majority of the work was produced using our new cell for combined *operando* X-ray diffraction and spectroscopy measurements [1]. This has been used extensively at the Swiss Norwegian beamline BM01B (now BM31) of the ESRF [2-5]. The presentation will also cover recent work using X-ray diffraction and pair distribution computed tomography carried out at beam line ID15a of the ESRF [6]. By combining *operando* synchrotron data with DFT calculations we have made significant progress towards understanding state of the art electrode materials for sodium ion batteries.

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3. Samarasingha, P.B., Sottmann, J., Margadonna, S., Emerich, H., Nilsen, O., Fjellvåg, H. Acta Materialia, 116, 290–297, 2016.
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Development of time-resolved X-ray absorption spectroscopy at SuperXAS beamline to study photocatalytic reactions

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⁵Department of Chemistry, University of Zurich, Switzerland

The setup for time-resolved X-ray absorption experiments in the nanosecond-microsecond time range has been developed at SuperXAS beamline of the Swiss Light Source [1]. It is optimized to measure weak signals from diluted samples with concentration <1mM. Detection system registers full kinetic information with 30 ns resolution without any delay scans. Method has been applied to multicomponent photocatalytic systems that reduce protons to H₂. Two intermediates of cobaloxime catalyst with Co(I) and Co(III)H centers have been observed in the microsecond range using time-resolved Co K-edge X-ray absorption spectra [2]. They have been characterized structurally on the basis of quantitative analysis of time-resolved XANES. We also studied the excited state structure of Cu photosensitizer. We found that blocking of access of solvent to the metal center is an efficient strategy to increase the excited state lifetime by a few orders of magnitude. Thus, we have demonstrated that the setup developed at the SuperXAS beamline of the SLS in combination with quantitative tools for XANES analysis are efficient to clarify mechanisms of the photocatalytic reactions and to develop photosensitizers with longer excited state lifetime.

[1] G. Smolentsev, A. Guda, M. Janousch, C. Friehe, G. Jud, F. Zamponi, M. Chavarot-Kerlidou, V. Artero, J. van Bokhoven and M. Nachttegaal, *Faraday Discuss.* **171**, 259 (2014)

[2] G. Smolentsev, B. Cecconi, A. Guda, M. Chavarot-Kerlidou, J. A. van Bokhoven, M. Nachttegaal, V. Artero, *Chem. Eur. J.* **21**, 15158 (2015)

Structure and properties of energy materials at SNBL and NcNeutron

Magnus H. Sørby, Christoph Frommen, Stefano Deledda, Bjørn C. Hauback

Institute for Energy Technology (IFE), Physics Department, Kjeller, Norway

Institute for Energy Technology (IFE) operates the Norwegian national infrastructure NcNeutron, which is a facility for neutron-based materials science connected to the reactor JEEP II. NcNeutron currently offers power neutron diffraction and small-angle neutron scattering, and instruments for neutron reflectometry and neutron imaging are in the design phase. The in-house research conducted by the Physics Department at IFE includes a wide range of materials for energy storage and conversion, such as hydrogen storage materials, battery materials, thermoelectrics, magnetocalorics and magnetic materials.

The complementarities between neutrons and synchrotron radiation are many. Besides the differences in contrast due to completely different interaction mechanisms, synchrotron radiation techniques have unsurpassed time-, space-, and angular resolution and signal-to-noise ratio. The possibilities to follow dynamical processes in-situ (BM01(A)) and obtain powder diffraction data with extreme resolution (BM01B/BM31) at SNBL has been particularly valuable in the studies of the above-mentioned materials.

Materials science is constantly moving to systems with increasing complexity with respect to compositions and structuring at different length scales, including disorder in 1, 2 or 3 dimensions. SNBL offers a complete suite of techniques to investigate the structure of matter from nearest atomic neighbors (XAS) to short- and long-range atomic order (total scattering and diffraction) and the nanometer range (SAXS through the agreements with DUBBEL). This talk will present our vision of further exploiting these techniques, in combination with advanced in-situ sample cells and non-synchrotron techniques such as Raman spectroscopy, in the future.

Metal hydrides for energy storage

Radovan Černý

Department of Quantum Matter Physics, Laboratory of Crystallography, University of Geneva, Quai Ernest-Ansermet 24, CH-1211 Geneva, Switzerland

The most promising approaches for advanced energy storage are based on direct storage of electricity in batteries and storage of hydrogen for later use in fuel cells. Complex hydrides based on light hydro- borate anions such as borohydride BH_4^- or *closo*-borane anion $\text{B}_{12}\text{H}_{12}^{2-}$ find their place in both applications, as solid stores for hydrogen, and since recently, also as solid electrolytes in Li- and Na-ion batteries.

X-ray powder diffraction applied in situ or in operando is one of the fundamental experimental techniques in search for novel materials and for studying the reactions and phase transitions related to storing hydrogen in solids and charging/discharging the batteries. The powder diffraction at synchrotron sources provides much higher time resolution and data quality compared to laboratory sources not only due to high photon fluxes but also due to recent development of 2D X-ray detectors.

In the talk, I will show how the equipment installed at the SNBL contributed to important progress in our research on solid electrolytes, and will give the perspectives of in operando studies of Na-ion batteries using borane based solid electrolytes.

X-Rays for Energy Materials

Prof. Dr. Andreas ZÜTTEL

LMER, ISIC, SB, École polytechnique fédérale de Lausanne (EPFL) Valais/Wallis, Energypolis, Rue de l'Industrie 17, CP 440, CH-1951 Sion, Switzerland

At the heart of the EPFL Valais Wallis project is a research institute in Sion with a major focus on renewable energy, sustainable chemistry, materials for environmental protection and also with a strong focus on analytical chemistry. There are seven research laboratories (<http://isic.epfl.ch/>) headed by professors who are also affiliated to the Institute of Chemical Sciences and Engineering (ISIC) at the main EPFL campus in Lausanne. Up to 200 researchers, comprising master project students, PhD students, postdocs and research fellows, are based in Sion. The focus of the research is on the interaction of surfaces, nano-porous and nano-structured materials with gases, e.g. hydrogen and CO₂ in order to adsorb gases, separate gases and for heterogeneous catalysis. The aim is to take advantage of the possibility to control materials composition and structures on an atomic scale in order to increase the interaction, use quantum phenomena and enhance selectivity in reactions. The materials of interest range from metal hydrides, metallic clusters, graphene, metallic nano-cubes and nano-carbons, metal organic frameworks (MOF's) to nanostructures on electrodes.

Our main focus on the SNBL is to perform in-situ experiments where structural changes and phase changes as well as the intercalation are investigated. Special equipment for the in-situ experiments has been developed, a high pressure, high temperature cell, the advanced gas analyzing system, the electrochemical X-Ray cell and a hydrogen compressor for safe operation of the high pressure experiments.

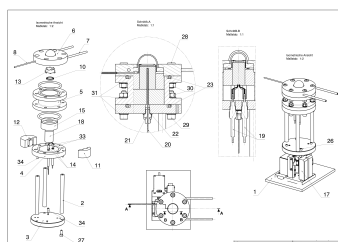


Fig. 1 High pressure (200 bar), high temperature (500°C) X-Ray cell

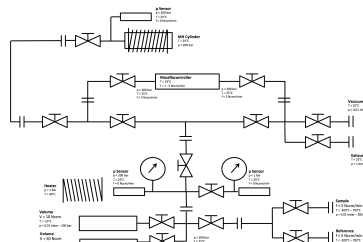


Fig. 2 Gas controlling and analyzing system



Fig. 3 High pressure hydrogen compressor (200 bar, 1000 bar)

An overview of recent developments and achievements will be given and the future perspectives will be discussed.

Biography



Born 22. 8. **1963** in Bern, Switzerland. **1985** Engineering Degree in Chemistry, Burgdorf, Switzerland. **1990** Diploma in Physics from the University of Fribourg (UniFR), Switzerland. **1993** Dr. rer. nat. from the science faculty UniFR. **1994** Post Doc with AT&T Bell Labs in Murray Hill, New Jersey, USA. **1997** Lecturer at the Physics Department UniFR. **2003** External professor at the Vrije Universiteit Amsterdam, Netherlands. **2004** Habilitation in experimental physics at the science faculty UniFR. President of the Swiss Hydrogen Association „HYDROPOLE“. **2006** Head of the section “Hydrogen & Energy” at EMPA and Prof. tit. in the Physics department UniFR. **2009** Guest Professor at IMR, Tohoku University in Sendai, Japan. **2012** Visiting Professor at Delft Technical University, The Netherlands, **2014** Full Professor for Physical Chemistry, Institut des Sciences et Ingénierie Chimiques, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland

Plans and Challenges in the Investigation of the Crystallographic and Magnetic Structures of Itinerant and Strongly Correlated Electron Systems

Diana Lucia Quintero Castro
University of Stavanger

Here, two scientific cases will be presented a multiferroic spinel and an itinerant magnet. Both electron systems are planned to be studied using X-ray methods within the SNBL program.

The spinel case is iron vanadate: FeV_2O_4 . It exhibits multiple magnetic and structural phases as a function of temperature. The geometrical arrangement of the Vanadium ions in a corner-shared tetrahedra induces magnetic frustration and this sums up to an orbital degeneracy that introduces complex orbital dynamics. The magnetic excitations at low temperatures (3K) show an anomalous broadening. The investigation of the unusual magnetic properties in this compound will be only possible if the nuclear structure is known. A high resolution single crystal X-ray diffraction experiment is planned for this compound.

The itinerant magnet case is ErPd_2Si_2 . In this compound a localized magnetic state coexist with a spin density wave from the conduction electrons, derived by the Fermi-surface nesting and/or their coupling to the local moments. It is unusual to find both contributions in a single magnetic system and it is an experimental challenge to distinguish the magnetic states related to each of them. Further X-ray diffraction experiments will be done in this compound in order to separate the contributions from the different orbitals.

Neutron diffraction and neutron inelastic scattering results will be presented in which the magnetic phases and excitations of these two systems are studied. Future experimental plans will be discussed as well as experimental challenges in X-ray techniques for the investigation of magnetic phenomena.

Bending magnet based CRG beam lines in the framework of ESRF's upgrade (EBS)

Axel Kaprolat

ESRF CRG-Liaison Engineer BP 220, F 38043 Grenoble Cedex France

Abstract

From 2018 onward the European Synchrotron Radiation Facility will undergo a machine upgrade by replacing the current storage ring by a completely new machine. Obviously, such a challenging project will have consequences for (both private CRG as well as public ESRF) experimental activities for around two years.

This talk will elaborate on the specifications of the new ESRF storage ring, the consequences for CRG beam lines in terms of facility shutdown and the overall schedule, the strategy for commissioning both the new storage ring and the beam lines accommodated at the ESRF and finally report on the post-EBS operating conditions for CRG beam lines.

Understanding bimetallic catalysts under working conditions by *in situ* combined XAS-XRD-MS

Sung Min Kim^a, Paula M. Abdala^a, Tigran Margossian^b, Davood Hosseini^a, Lucas Foppa^b, Andac Armutlulu^a, Wouter van Beek^c, Aleix Comas-Vives^b, Christophe Copéret^b and Christoph Müller^{a,*}

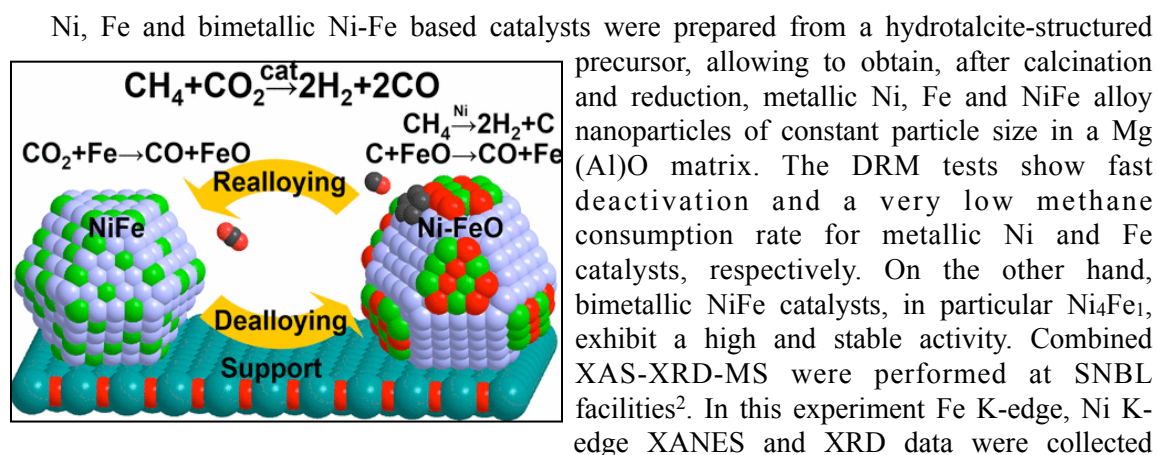
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The dry reforming of methane, DRM ($\text{CH}_4 + \text{CO}_2 \rightarrow 2\text{CO} + 2\text{H}_2$) is a promising process to convert two greenhouse gases into a synthesis gas, a useful chemical feedstock. Ni based catalysts are attractive for this process because they combine good activity and low costs compared to noble metals. However, these catalysts suffer from rapid deactivation due to metal oxidation, particle sintering and coke formation. Alloying nickel with other noble or non-noble metals can improve the catalytic performance in terms of activity and stability. However, the underlying mechanism that explain the improved activity and stability of Ni based bimetallic systems is a topic of debate. Here, we present a *in situ* combined X-ray absorption spectroscopy, X-ray powder diffraction and mass spectrometry (XAS-XRD-MS) study to obtain understanding at the atomic level on how the addition of Fe affects the performance of Ni-Fe based DRM catalysts.¹



consecutively, while the off-gas was monitored by a mass spectrometer. These experiments allowed us to monitor the changes in the oxidation state and structure of the catalyst during DRM reaction. Moreover, the results were correlated with *ex-situ* characterization techniques (such as Raman spectroscopy, TEM, STEM-EDX) and DFT calculations. These experiments allowed us to understand the dynamic of the NiFe system under reaction conditions and to address the cooperative role of Ni and Fe that lead to a stable performance for DRM. Under DRM, Fe is oxidized partially to FeO leading to a partial dealloying and formation of a Ni-rich NiFe alloy. Fe migrates leading to the formation of FeO preferentially at the surface. Experiments in an inert helium atmosphere confirm that FeO reacts via a redox mechanism with carbon deposits forming CO, whereby the reduced Fe restores the original Ni-Fe alloy.

References:

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2. W. van Beek, O. V. Safonova, G. Wiker and H. Emerich, *Phase Transitions*, 2011, **84**, 726-732.

Nanomaterials for Catalysis

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¹ Centre for Materials Science and Nanotechnology, Department of Chemistry, University of Oslo, POBox 1033, N-0315 Oslo, Norway

² ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire OX11 0QX, United Kingdom

Abstract

Various types of model materials for catalysis are synthesized in the UiO home laboratory and studied ex-situ and at operando conditions at SNBL. The systems investigated range from mono/bimetallic nanoparticles (Co; Co-Re; Pt-Rh; Pt-Pd etc.) to catalysts where such NPs are investigated on mesoporous supports. Issues of interest include clarification of solid solution situations versus core shell situations for the NPs, polytypes/polymorphs of cobalt relative to synthesis/temperature/atmosphere conditions and description of various stacking variants, furthermore careful studies of layered double hydroxides as supports and their stacking disorder with total scattering analysis as tool for discriminating between ordered, partly disordered and randomly stacked layered materials without 3D correlations. The NP model studies are part of larger efforts where reactor STM is used for surface dynamic studies at operando conditions.

XAFS, XRD and PDF at SNBL: application to catalytic materials in operando at a unique resource

Mark A. Newton,¹ Jeroen A. Van Bokhoven,¹ Davide Ferri,² Christophe Coperet,¹ Mimi Hii³

¹ *ETH Zurich, Switzerland*

² *PSI, Villigen, Switzerland*

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Examples of the application of quick EXAFS, and Total X-ray scattering/ PDF to three distinct areas of operando materials chemistry and catalysis will be given to demonstrate both the versatility and unique nature of BM31 at the ESRF in addressing issues of importance to Swiss research groups at PSI and ETH, Zurich. These will include: a novel source of leaching of Pd in heterogeneous (liquid-solid) systems for heterogenised carbon carbon coupling chemistry; investigating the active site in Cu/Zeolite reactive materials for methane to methanol conversion; and seeing into the mechanism of MOF crystallisation using combined EXAFS/PDF.

These examples will be used to highlight the strategic importance of SNBL and a foundation for considering its maintenance and future development.

In-situ toolbox for studying nucleation and growth in oxide thin films and hierarchical structures prepared from aqueous solutions

Anders Bank Blichfeld, Department of Materials Science and Engineering, NTNU Norwegian University of Science and Technology

High brilliance and small beam divergence are a requirement to gain new insight to electronic and mechanic materials where the dominant mechanisms are governed by features on the Ångström-and nano-scale. In a recently started Toppforsk project, From Aqueous Solutions to oxide Thin films and hieratical Structures (FASTS), we aim to develop an aqueous synthesis platform for thin films and hierarchical structures based on an *in-situ characterization toolbox*. Aqueous chemical synthesis methods are environmental friendly and highly flexible routes to tailored nanostructures and will serve as the basis for this project focusing on lead-free piezo- and ferroelectric materials.

A high brilliance X-ray source opens up for sub-second time resolution for chemical reactions in even dilute systems that is required to obtain a high control and reproducibility. Understanding the kinetics in for example hydrothermal synthesis will make it possible to tune the parameters for obtaining metastable phases. Combining this with theoretical principles and models, will enable precise control of nucleation and growth of the oxide nanomaterials and replace current empirical approaches.

The FASTS project is focused on developing two synchrotron XRD setups: an in-situ hydrothermal synthesis cell that is a close to the experimental setup in laboratories that can easily be scaled up for the industrial production, and an *in-situ* cell with atmospheric control for the pyrolysis step in chemical solution deposition processing of thin films.

Using XAS to elucidate the biogeochemistry of trace elements in environmental systems

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Trace elements have wide-ranging global impacts on environmental and human health due to their dual role as essential nutrients and toxic contaminants. Understanding the biogeochemical cycling and fate of trace elements in natural and technical systems is therefore of primary importance. In this context, knowledge on the speciation of trace elements is pivotal because it controls the solubility and thus the mobility and bioavailability of trace elements. Synchrotron-based X-ray absorption spectroscopy (XAS) provides element-specific molecular-level insight into the oxidation state and coordination of trace elements in complexed, amorphous and crystalline form and allows to quantify the fractions of different trace element compounds in complex environmental matrices such as soils and sediments, ideally down to trace concentrations of a few or few tens of mg/kg. In this presentation, the use of XAS for the elucidation of trace element speciation and dynamics will be discussed based on ongoing work concerned for example with the transformation of engineered Ag and Cu nanoparticles in urban sewer systems or the speciation of trace elements in contaminated soils. The examples serve to highlight the importance of XAS to advance the quantitative and mechanistic understanding of environmental processes and to address needs for the effective use of XAS in environmental trace element biogeochemistry.

Investigation of the host-guest chemistry in coordination polymers using in-situ and operando experiments

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The quality of radiation and specialized experimental setups at large-scale synchrotron radiation facilities enable researchers to perform experiments impossible or difficult to implement in their home laboratories. In respect to X-ray diffraction experiments, the increased intensity of the beam generated at the synchrotron generally allows faster data collection with better signal-to-noise ratio of both single crystal and powder samples. On the other hand, limited stability of samples in the beam might pose problematic. Experimental setups like high-resolution powder diffractometers permit clearer assignment of reflections which benefits indexation, structure solution and refinement. The combination of high intensity and fast detectors nowadays available at synchrotron facilities also make possible diffraction experiments in which fast and/or dynamic processes are monitored in-situ or in-operando where time resolution and data quality are sufficient to gain a detailed picture about the process. In the best case, structural information can be gained from each diffraction pattern collected, thus revealing a complete picture of the processes occurring in the material.

This presentation will present examples from the group's research where experiments at the Swiss-Norwegian Beamlines at the ESRF have helped determine the crystal structure of solvated and desolvated coordination polymers, observe chemical and structural transformations upon solvent removal and permitted the determination of the crystal structure of host-guest compounds identifying the adsorption site(s) on the framework.

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INHIBITION OF CLASS 1A RIBONUCLEOTIDE REDUCTASE, AND A COMPARISON OF THE DIMANGANESE ACTIVE SITES OF CLASS IB RIBONUCLEOTIDE REDUCTASE AND MANGANESE CATALASE

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Ribonucleotide reductases (RNRs) are enzymes that convert RNA building blocks into DNA building blocks [1]. The reductive reaction by RNRs requires a cysteine thiyl radical, which, in the case of class Ia or Ib RNRs, is initiated by an Fe^{III}₂- or Mn^{III}₂-tyrosyl radical (Y•) cofactor in the R2 subunit of RNR. During enzymatic turnover, the cofactor is activated by oxygen, and generates a Y• that is transported from the smaller R2 subunit to the large catalytic subunit R1 of RNR, where DNA building blocks are formed.

The small subunit of class Ia RNRs can be inhibited by several small compounds [2,3], through the inhibition of the active Fe^{III}₂- Y• cofactor. We have performed interaction studies and K_d measurements of a mammalian R2 protein with several newly synthesized compounds, and studied their potential inhibitory effect on the protein with EPR, showing promising results.

Manganese catalase (MnCAT) enzymes [4,5,6] contain an active site that is similar in structure to the Mn^{III}₂ form of NrdF (the R2 subunit in class Ib RNR), characterized by a carboxylate-bridged Mn^{III}-O-Mn^{III} cofactor. However, it catalyzes a different reaction – the degradation of hydrogen peroxide to dioxygen and water. A still unresolved question is how these enzymes containing similar active sites can catalyze different reactions. A variety of spectroscopic methods have been used to try to resolve this question. Samples containing NrdF with active Mn^{III}-O-Mn^{III} cofactor have been prepared and studied by circular dichroism (CD) and magnetic CD (MCD) spectroscopy [6]. The data show both similar and distinct features as compared to MnCAT.

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High-resolution 3D imaging at the cSAXS beamline using X-ray ptychographic tomography

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Abstract

X-ray ptychography is an imaging technique in which the specimen is scanned across a finite, coherent X-ray beam and diffraction patterns are recorded in the far field at each scanning position such that the illuminated areas partially overlap. Using iterative phase retrieval algorithms, an image of the sample's transmissivity can be reconstructed, providing quantitative absorption and phase contrast. The resolution in X-ray ptychography is not limited by the beam size or by the scanning step, but by the largest angle at which diffraction patterns can be reliably measured, although in practice positioning stability and radiation damage can also limit the resolution. At the coherent small-angle X-ray scattering (cSAXS) beamline at the Swiss Light Source we have successfully implemented X-ray ptychography combined with tomography to provide a tool for 3D imaging on the nanoscale with quantitative density contrast. The imaging quality of this technique is exploited by many users from different research areas such as chemistry, materials science and biology. The upcoming diffraction-limited storage rings will provide about a factor 50 higher coherent flux, offering opportunities for higher resolution, larger volumes and/or faster measurements.

In this presentation I will show examples of current applications of ptychographic tomography and will give some perspectives for its use in future synchrotron sources.

Combining Protein X-ray Crystallography and *in situ* Single-Crystal UV-Vis and Raman Spectroscopy to Grasp the True Structure of Haem- and Flavoproteins

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X-ray crystallography is a central method for studying the structure-function relation of redox proteins, however the crystal structures are missing key information. This includes oxidation state, protonation and spin state, which are essential for understanding the reaction mechanisms of these redox protein systems. Therefore, a combination of crystallographic and spectroscopic methods is vital for a more fully characterisation. Additionally, redox proteins containing different cofactors as haem and flavin groups are very labile for X-ray induced radiation damage resulting in reduction of the cofactors during crystallographic data collection. For these systems UV-Vis and Raman spectroscopy can be used to monitor the radiation damage. Single-crystal *in situ* spectroscopy setups at different synchrotron beam lines have been utilized, and we have developed a combined UV-Vis and Raman setup at the Swiss-Norwegian Beam Lines at the European Synchrotron Radiation Facility. Several haem- and flavoproteins have been studied resulting in complementary structural information.

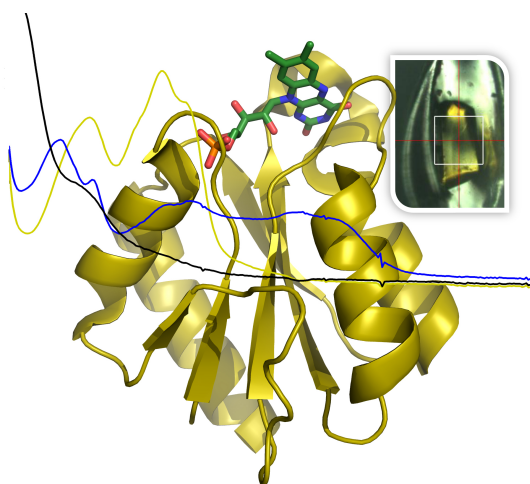


Figure 1: Structure of a flavin protein with crystal and UV-Vis spectra

Skin penetration study of drug carriers using soft X-Ray spectromicroscopy

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Abstract

The aim is to achieve effective drug concentrations directly at the site of the disease. Small carrier systems, called nanocarriers, are loaded with active substances. The special research area investigates the basic mechanisms of topical therapy using nanocarriers in inflammatory skin diseases. The skin is suitable because of its easy accessibility since the transport paths is short and the controlled released of the active substances by internal as well as external controllable stimuli. The steps from the chemical synthesis of nanocarriers to the foundations of topical delivery are to be optimized for this purpose. This optimization will develop nanoscopic carriers systems with deliberately refined properties which enable improved concepts for the topical therapy of inflammatory skin diseases which are associated with a barrier of the skin. The focus of the work is to develop a nanocarrier with a size of 10-100 nm, which mainly consist of biocompatible architectures. We are proposing selected anti-inflammatory active ingredients incorporated into these nanocarriers and then in a controlled manner by using selective triggers such as temperature or pH. The absorption contrast gives direct information about electron density and it's easy to interpret without any image reconstruction process required for phase contrast imaging. Soft X-ray microscopy is suitable for high resolution observation of a whole cell in a thickness of a few μm .

Soft Matter research at SNBL and NcNeutron

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Institute for Energy Technology (IFE) operates the Norwegian national infrastructure *NcNeutron*, which is a facility for neutron-based materials science connected to the reactor JEEP II. *NcNeutron* currently offers power neutron diffraction and small-angle neutron scattering, and instruments for neutron reflectometry and neutron imaging are in the design phase. One important part of the in-house research conducted by the Physics Department at IFE is within the field of soft matter, such as polymer gels and networks, colloidal systems and magnetic fluids.

During recent years we have seen a rapidly growing research activity in this area - in Norway and worldwide - covering a broad range of topics. The development is driven by the search for lightweight materials with advanced functionalities as well as the need to obtain fundamental insight into the organization and dynamics of non-crystalline matter.

This research depends critically on access to different types of small-angle scattering (SAS) instrumentation, and new x-ray/neutron infrastructure projects in Europe and elsewhere generally include one or more SAS setups as a primary element. The soft matter group at IFE make heavily use of the small-angle neutron scattering setup (SANS) located at the JEEP II reactor at Kjeller, but also benefits from access to synchrotron x-rays at SNBL as well as the SAXS setup available to Swiss and Norwegian users at BM26 (ESRF) through the collaboration agreement with SNBL. This talk will give examples of ongoing research, and give some perspectives for the future and needs for access to advanced synchrotron-based experiments.

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