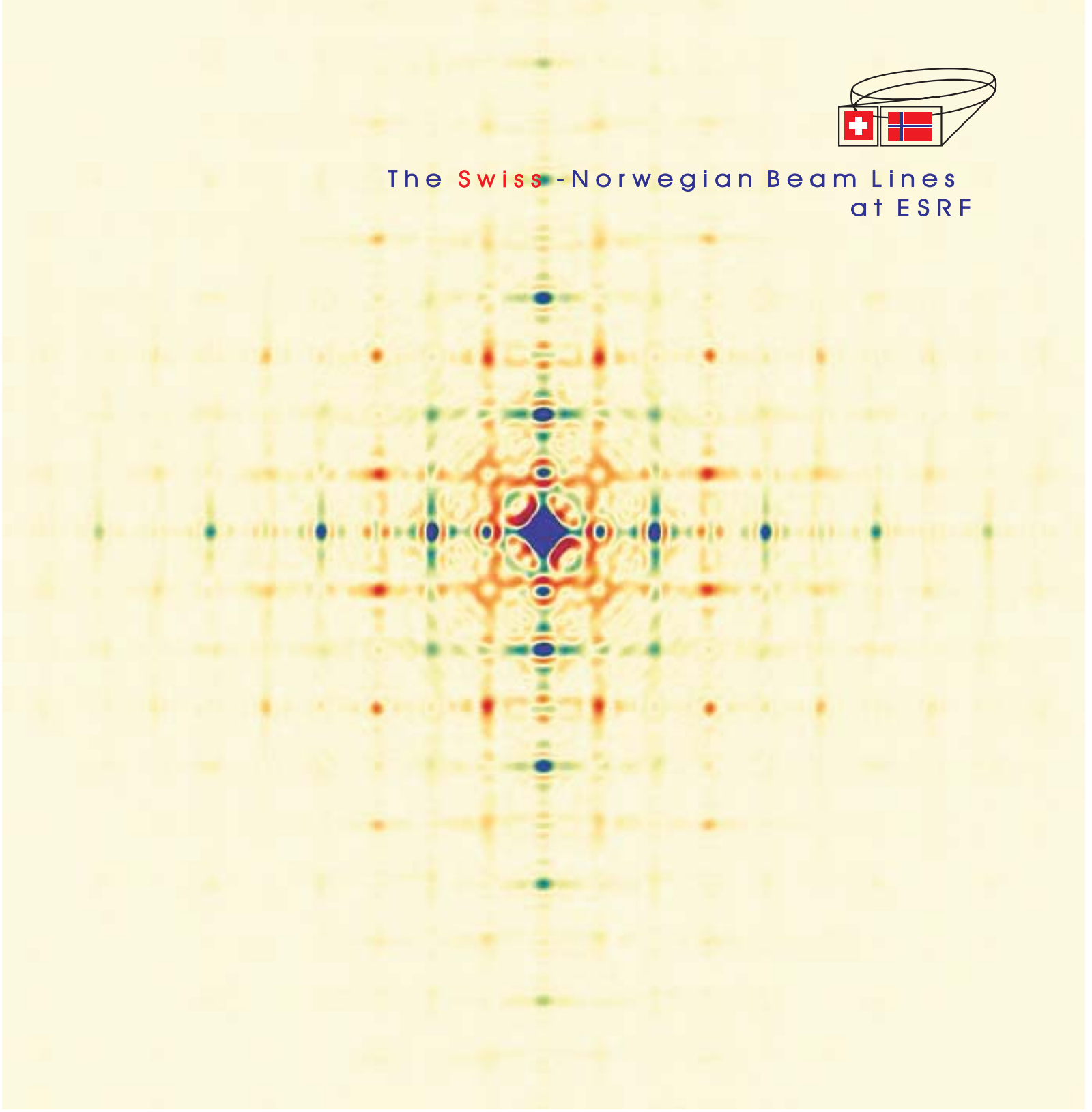

The Swiss-Norwegian Beam Lines at ESRF

09/10

ACTIVITY REPORT

CONTENTS

INTRODUCTION

The two years covered by this report have seen an unprecedented increase in the number of papers which have been published using data collected at SNBL. A total of well over two hundred peer-reviewed publications are listed in this report for the period 2009-2010. We have also recently passed the milestone of one thousand papers published since the beginning of operation of the Swiss-Norwegian beamline. The management of SNBL would like to take this opportunity to thank our staff for all their efforts over the years, and to congratulate our user community for their extraordinary level of scientific output. The successful mix of scientific techniques available on the Swiss-Norwegian beamline has made SNBL into one of the most productive beamlines at the ESRF.

Following the signing some years ago of a Memorandum of Understanding (MoU) with the Dutch-Belgian beamline at the ESRF, we are pleased to announce that a further collaboration agreement has been established between SNBL and MaxLab. The new MoU regulates the technical, scientific and administrative areas to be covered by this collaboration, including the exchange of in-house research beamtime, the possibilities of staff exchanges and visits between the two institutes, and the development of new equipment which may be of common interest. SNBL has already been host to visiting scientists from MaxLab, and we look forward to continuing this fruitful collaboration.

After the extensive upgrade of the infrastructure of the beamline during 2007 and 2008, the last two years has been both a time of consolidation for SNBL and also of preparation for some major new investments. In line with the recommendations of the Committee of the Future presented in November 2008, the management of SNBL has put forward a roadmap for investment in new detectors for diffraction experiments and in improving the optics for EXAFS. Grant requests totaling about €1M for a new large area detector and associated equipment have been submitted to the Swiss National Science Foundation and to the Norwegian Research Council. The Swiss NSF has already reacted favorably, and the result of the grant request to the NRC is still pending. The SNX Council has also approved spending on a new CCD detector for the KM6 diffractometer, and investment will shortly begin on new optics for the EXAFS. The management and staff of SNBL are firmly committed to maintain and improve the scientific capabilities of the beamline, and we are grateful both to our national research agencies and to the SNX Council for their continuing support and for the provision of the necessary financial resources.

V. DMITRIEV, P. PATTISON, H. EMERICH

SCIENTIFIC HIGHLIGHTS

Pressure-induced insulator-to-metal transition in $TbBaCo_2O_{5.48}$

Principal publication and authors
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A key aspect of cobalt oxides that eclipses the conventional charge, spin and orbital degrees of freedom, is the possible variety of spin states of the cobalt ions. For this reason, cobalt layered perovskites represent a unique system that allows the study of the effect of spin-state degrees of freedom on the metal-insulator charge- and spin-ordering transitions.

The complex oxide $TbBaCo_2O_{5.5}$ contains Co^{3+} ions in octahedral or pyramidal coordination of oxygen atoms. Each Co^{3+} ion can be found in a low, intermediate, or high spin state (LS, IS, or HS) having different ionic radii. $TbBaCo_2O_{5.5}$ shows an insulator-to-metal transition on heating, and at the same temperature, T_c~335K, there is a structural change from Pmma ($2a_c \times 2a_c \times 2a_c$) to Pmmm ($a_c \times 2a_c \times 2a_c$). This transition agrees with a partial ordering of cobalt ions in different spin and orbital states. The deactivation of a "spin blockade" mechanism (associated with the LS states) has been suggested for this insulator-to-metal transition, assuming that heating would promote HS states and suppress LS states [1].

There are several reasons to also expect a change in structural and transport properties of insulating oxides of 3d-metals as a function of pressure. Pressure could favour the orbitally-disordered phase thus giving an increase of conductivity. A change of spin-state for cobalt ions from HS or IS to LS is accompanied by a decrease of ionic radius, thus pressure could favour the spin conversion toward LS states. In this case one would not expect an increase of conductivity under pressure since LS states effectively block electron mobility via the "spin blockade" mechanism. One more possible pressure effect may be foreseen from symmetry analysis that links the structure and conductivity via an electron transfer integral through the Co-O-Co path. For this mechanism, one would expect a symmetry change under pressure.

As no change in physical and structural properties has been found so far between ambient pressure and 1.2 GPa [2], the "spin blockade" has been considered the primary mechanism for the temperature induced metal-to-insulator transition in rare earth cobaltites.

By combining diffraction and transport measurements, we have recently shown that there is an insulator-to-metal transition, but at pressures higher than probed before, at ~10 GPa (Figures 6 and 7). In the same pressure range we observe changes in resistivity and in the ratio of the lattice constants of the average orthorhombic Pmmm ($a_c \times 2a_c \times 2a_c$) cell; the same behaviour holds for the spontaneous strains. The change in the ratio of the lattice constants is very similar to that observed as a function of temperature (Figure 7), thus suggesting a common mechanism for the two transitions. We can exclude the spin blockade mechanism as candidate, as it should be suppressed under pressure due to the higher ionic radius of HS ions. Temperature favours disorder

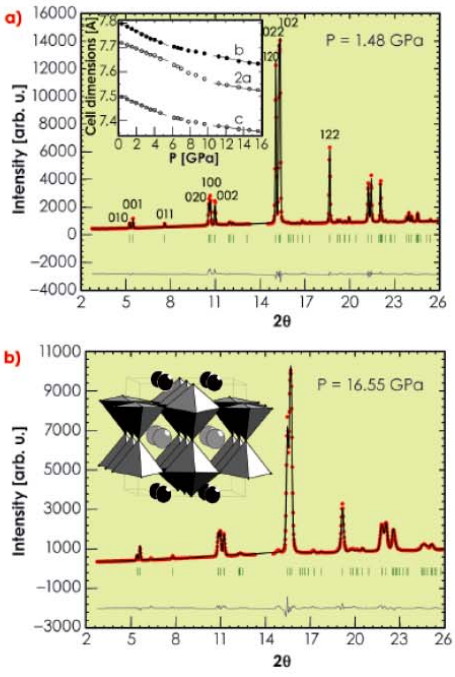

Fig. 6: Diffraction patterns for $TbBaCo_2O_{5.48}$ with experimental points, fitted profile, difference curve (bottom), and positions of Bragg reflections (vertical bars). a) P=1.48 GPa; the inset shows the unit cell dimensions as a function of pressure. b) P=16.55 GPa; the inset shows the high temperature/high pressure orthorhombic Pmmm ($a_c \times 2a_c \times 2a_c$) structure. Colour code: Tb (black), Ba (grey), cobalt ions are coordinated by oxygen forming octahedrons and pyramids.

via the entropy term, but pressure does not; ordering of spin states can therefore be excluded as well as common mechanisms. We propose instead that the change of the Co-O-Co angle affects the electron transfer integral, and consequently the conductivity. This mechanism assumes a strong link between structural deformation and transport properties while the spin-state degree of freedom only plays a secondary role.

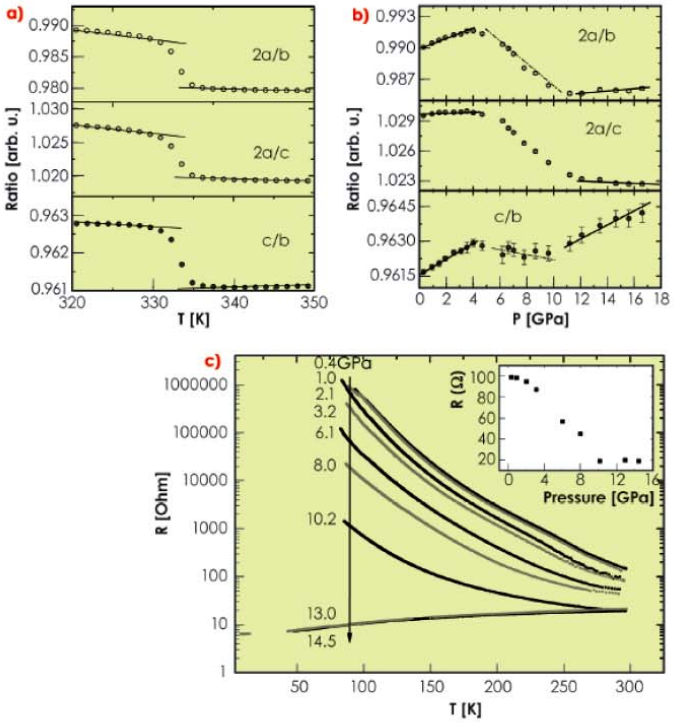

Fig. 7: Ratio of unit cell dimensions as a function of a) temperature, and b) pressure. c) Temperature dependence of the resistivity measured at different pressures. The inset shows the pressure dependence of the resistivity at 298 K.

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Structure and properties of single crystals of high-pressure boron

Boron is an unusual element. Its three valence electrons are too localised to make it metallic but insufficient in number to form a simple covalent structure. As a compromise, boron atoms form quasimolecular B_{12} icosahedra that become building blocks of boron and boron-rich crystalline phases. Only α-rhombohedral and β-rhombohedral boron, obtained at ambient pressure, are established as pure boron crystalline forms. The high-pressure behaviour of boron is of considerable interest in physics and materials science. Our investigations of superconducting polycrystalline boron-doped diamonds [1] revealed boron segregation in an intergranular space that may influence the mechanism of superconductivity.

Many of the fundamental questions regarding the solid-state chemistry of boron are still unanswered. Synthesis of single-phase products in macroscopic quantities and in the form of single crystals remains one of the great challenges of boron chemistry [2]. Using a large volume press, at 20 GPa and 1700 K, we have grown single crystals of a high-pressure high-temperature orthorhombic B_{28} boron phase for the first time. The crystals appeared to have a dark-red colour and an elongated prismatic shape (Figure 59). We confirmed the purity of the crystals by chemical analysis, collected single-crystal X-ray diffraction data (BM01), then solved and refined the crystal structure. There is remarkable

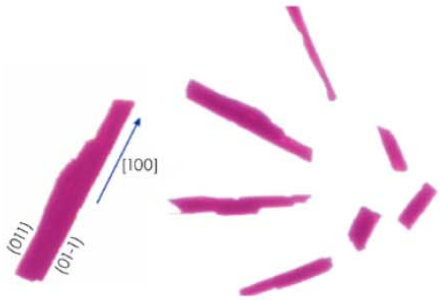

Fig. 59: Single crystals of the orthorhombic high-pressure boron phase.

Principal publication and authors
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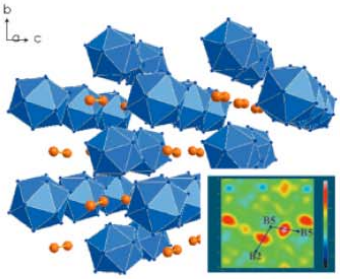

Fig. 60: The structure of the HPHT boron phase shown in the *bc* projection. The insert shows difference electron density plots around the atoms B5 (dumbbell, upper right) and B2 and B5 (dumbbell-icosahedron contact) extracted from single-crystal X-ray diffraction data. The maximum electron density is centred on the middle of the bond, suggesting covalent bonding between the B_2 dumbbell and the B_{12} icosahedron.

agreement between structural parameters obtained from single crystal and powder X-ray diffraction data [3] and those from our *ab initio* calculations. The *bc* projection (**Figure 60**) shows that the structure is built of B_{12} icosahedra and B_2 dumbbells linked together, thus forming a three-dimensional network. The distances B-B within a B_{12} icosahedron (1.766(3)-1.880(3) Å) are slightly longer than those within a dumbbell (1.721(4) Å). Chemical bonds within B_2 dumbbells and B_{12} icosahedra, as well as between icosahedra and dumbbells, are strongly covalent, as evident from the presence of residual electron density in the difference Fourier maps.

The high density and strong covalent bonding in this B_{28} phase suggest that it could be less compressible than the other known boron phases. This was confirmed in the compression experiment (**ID09**) up to ~30 GPa in a diamond anvil cell with Ne as a pressure transmitting medium and a powder of B_{28} that gave values for the bulk modulus of $K_{300} = 227(2)$ GPa and its pressure derivative $K' = 2.2(2)$. Pure orthorhombic B_{28} is a poor electrical conductor with a resistivity of the order of 10^6 Ω cm at ambient conditions. With increasing temperature, the resistivity decreases indicating semiconducting behaviour. The activation energy (1.9(2) eV) is in reasonable agreement with the value of the band-gap energy (2.1 eV) determined from optical spectroscopy measurements.

Boron is known as a hard material (with the Vickers hardness reported as high as 25-30 GPa for β-boron). We found that samples of B_{28} with submicrometre grain sizes have a hardness of 58(5) GPa. This value is in the range of polycrystalline cBN which makes B_{28} the second (after diamond) elemental superhard material.

In summary, our study demonstrates that the orthorhombic high-pressure high-temperature boron phase, synthesised above 9 GPa, has a structure consisting of covalently bonded B_{12} icosahedra and B_2 dumbbells, and combines unusual properties – it is a wide band gap semiconductor that is superhard, optically transparent, and thermally stable (above 1000 K in air). We have demonstrated the possibility to grow single crystals of this phase, which opens prospects for applications of this material in areas of electronics and optics.

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Principal publication and authors
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Consequences of using X-rays when studying redox cofactors

Flavin cofactors are involved in numerous biological processes including light sensing, oxygen activation, electron transfer, and DNA-repair. During these processes the flavin cofactor undergoes subtle conformational changes depending on its state. It is of great importance to understand the connection between flavin conformations and the nature of the chemistry performed.

In the literature, questions regarding flavin structure and function have been addressed many times using spectroscopic methods, theoretical calculations, and X-ray crystallography. The protein data bank (PDB) contains more than 1400 crystal structures containing either a flavin mononucleotide (FMN) or flavin adenine dinucleotide (FAD) cofactor. The redox and protonation states of these cofactors are usually assumed to reflect the situation in the protein crystal prior to X-ray data collection. This is a problematic assumption since redox cofactors are susceptible to react with photoelectrons generated in the crystal by X-rays during data collection. The large distribution of the so-called flavin butterfly bending angle, which is an indicator of flavin red-ox and protonation state, in the PDB-deposited flavoproteins indicate a large diversity of flavin states among these structures [1].

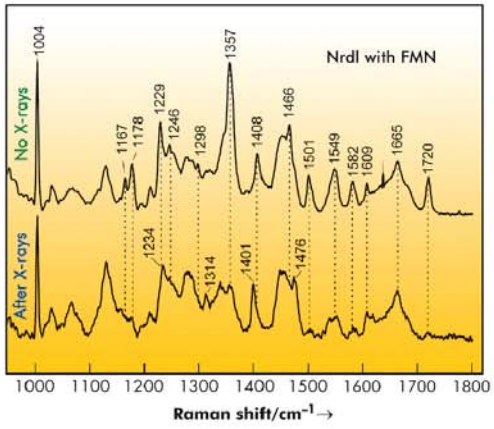

Fig. 1: Single-crystal *in situ* Raman spectra recorded for the flavoprotein NrdI before and after X-ray data collection.

We have solved crystal structures of the flavodoxin-like protein NrdI to 1.12 and 1.15 Å resolution at beamline **BM01** using crystals containing the yellow, oxidised FMN ($NrdI_{ox}$) and the blue, neutral semiquinone FMNH• cofactor ($NrdI_{sq}$), respectively. Before and after X-ray data collection, Raman spectra were collected *in situ* from both crystals. For both crystals, Raman bands confirmed the initial redox states. However, as shown in **Figure 1**, dramatic changes in the Raman spectra recorded before and after X-ray data collection

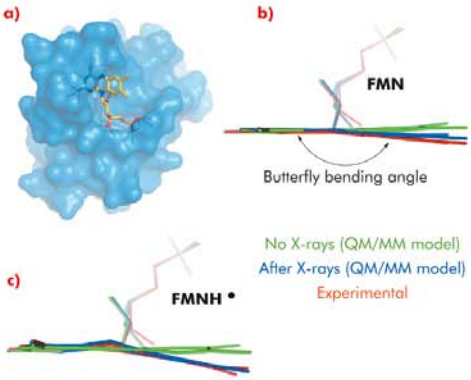

Fig. 2: a) Stick representation of the flavin cofactor bound in a surface pocket of NrdI, b) QM/MM models of flavins in NrdI containing the oxidised FMN cofactor (green), the one-electron reduced ($FMN^{\bullet -}$) cofactor (blue), and the flavin conformation in the crystal structure of $NrdI_{ox}$ (red), c) QM/MM models of flavins in NrdI containing the neutral semiquinone ($FMNH^{\bullet}$) cofactor (green), the one-electron reduced ($FMNH^{-}$) cofactor (blue), and the flavin conformation in the crystal structure of $NrdI_{sq}$ (red). In both b) and c) the QM/MM models confirm a one electron reduction of the flavin during X-ray data collection.

were observed. When starting with $NrdI_{ox}$ all flavin Raman bands either shift in energy or disappear after X-ray data collection, indicating a change in the flavin state. For $NrdI_{sq}$ all Raman bands decrease in intensity upon X-ray exposure, indicating a one-electron reduction to the fully reduced anionic form $FMNH^{-}$. These observations coincide with the crystal structures in which the flavin in both models have a butterfly bend deviating from the planarity predicted for FMN and $FMNH^{\bullet}$.

To shed light on our experimental observations, which indicate that the state of the flavin cofactor is influenced by exposure to X-rays, QM/MM models of possible flavin redox and protonation states were built and their geometry optimised. Of the different models containing FMN, $FMN^{\bullet -}$, $FMNH^{\bullet}$, $FMNH^{-}$, or $FMNH_2$, the one-electron reduced forms of the initial states in the crystals, $FMN^{\bullet -}$ and $FMNH^{-}$, corresponded with the experimentally determined ($NrdI_{ox}$) and ($NrdI_{sq}$) crystal structures respectively.

The combination of single-crystal *in situ* Raman crystallography with QM/MM calculations demonstrate the importance of monitoring active site cofactors during X-ray data collection in order to track and characterise eventual radiation damage, which, if unaccounted for, can result in erroneous interpretation of crystal structures [2,3,4,5].

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Flexible porous coordination polymers for selective adsorption

Among crystalline porous solids, metal organic frameworks (MOFs) deserve special interest. These hybrid solids, built up from inorganic subunits connected through polytopic ligands (such as poly-carboxylate and poly-pyrazolate) have the faculty to change their pore size reversibly upon external (temperature, pressure, light) or internal (entrapped guests) stimuli while remaining crystalline. This dynamic porosity leads to unusual sorption properties, which are attractive for applications in the area of fluid storage, capture or separation. One of the most archetypical examples of such a solid is MIL-53(M) (MIL stands for Materials Institute Lavoisier), a metal (M = Al, Cr, Fe) terephthalate built up from inorganic chains and linear ligands presenting one dimensional diamond-shaped micropores. In the last few years, a multi-technique approach has been developed to study the adsorption of important gases (H_2, CH_4, CO_2, alkanes, etc.) in these solids [1].

The sorption of polar vapours (water, methanol and ethanol) in MIL-53(Cr) has recently been explored. Upon adsorption, the solid evolves from a large-pore form to a narrow-pore form (**Figure 1**), for which the pore volume depends on the nature of the guest. At higher alcohol pressure, the structure evolves once again to a large-pore form, whereas the narrow pore form is retained in the presence of water. The specific host-guest interactions associated with these adsorption processes were probed by microcalorimetry, X-ray structure investigation, IR spectroscopy, thermal analysis and molecular modelling. The in situ powder X-ray diffraction and Raman experiments were carried out at beamline **BM01A**. These studies revealed that the strength of the interaction depends on the nature of the adsorbed species (water < alcohols), and finally concludes that this solid is potentially interesting for the selective adsorption of alcohol from

Principal publications and authors
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T. Devic (d), P. Horcajada (d), C. Serre (d), F. Salles (c), G. Maurin (c), B. Moulin (b), D. Heurtaux (d), G. Clet (b), A. Vimont (b), J.-M. Grenèche (e), B. LeOuay (d), F. Moreau (d), E. Magnier (d), Y. Filinchuk (f), J. Marrot (d), J.-C. Lavalley (b), M. Daturi (b) and G. Férey (d), J. Am. Chem. Soc. **132**, 1127-1136 (2010).
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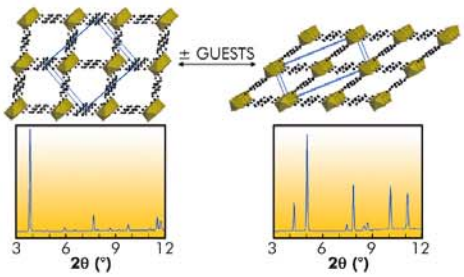

Fig. 1: The "breathing" behaviour of MIL-53 upon adsorption/desorption of guests, and the corresponding powder X-ray diffraction patterns which allow a direct evaluation of the pore size. Left: large-pore form; right: narrow-pore form.

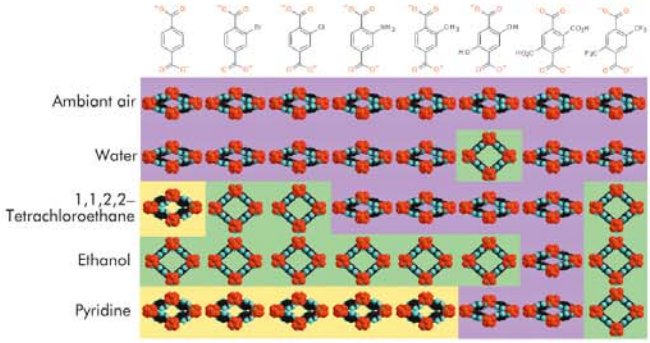

Fig. 2: Effect of the nature of the grafted organic group on the pore opening of functionalised MIL-53(Fe)s upon adsorption of various liquids. Pink: narrow-pore form; green: large-pore form; yellow: intermediate-pore form.

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water/ethanol mixtures, which could be of interest in industrial applications to obtain alcohol of very high purity. Furthermore, the introduction of functional groups on the organic linker is an easy way to modify the pore surfaces, and thus to tune the sorption properties, affecting both the strength of the host-guest interactions and the pore opening. With this aim, various functionalised MIL-53(Fe) solids were prepared. While the hydrated form (under ambient air) of all these solids is associated with a narrow-pore form (see **Figure 2**), they present contrasting behaviours when immersed in liquids.

Powder X-ray diffraction indeed reveals that the pore opening (and thus the number of adsorbed molecules), strongly depends on the nature of both the liquid and the grafted group (**Figure 2**), and that the appearance of a large-pore form requires that the host-guest interactions (hydrogen bonding, interactions between grafted functional groups) overcome the intraframework interactions. Providing that this requirement is fulfilled, the adequacy of the chemical nature of both components (liquids-grafted groups) could lead to selective adsorption.

Focus on: nature and energy

Fuelling nature to pr

Transportation fuels that reduce emissions are a promising answer to our polluted world. Despite its discovery in the 1920s, Fischer-Tropsch catalysis may well be a good candidate for a cleaner future. Colourless, odourless and low in toxicity, Fischer-Tropsch-based diesel fuel reduces sulphur, nitrogen oxide and carbon monoxide emissions compared to regular diesel, and it does not require engine modifications in vehicles for its use.

Image of the gas to liquids plant of Shell in Malaysia xxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx

Hydrocarbons, such as crude oils, bitumen and natural gas, are still available in nature, so Fischer-Tropsch (FT) synthesis has only been partially popular since its discovery back in 1925. In certain moments in history it became relevant when there was a lack of easily available crude oil: during World War II its use was mostly military, later, the oil crisis in the 1970s led to some recovery of interest in FT. Today, the fact that natural liquid hydrocarbons are becoming less abundant, not easily accessible and containing more impurities, such as sulphur, nitrogen and metals, and that there is an increasing demand for a more sustainable society, has put FT back in the limelight.

FT technology converts natural gas, coal or biomass to clean-burning fuel or hydrocarbons. In a first step, these resources are converted to synthesis gas, in short syngas (mixtures of carbon monoxide and hydrogen). Then the syngas reacts on a cobalt or iron catalyst to yield hydrocarbons in a kind of polymerisation process, leading to mainly long-chain hydrocarbons or waxes. These are often broken up again in diesel fuel molecules.

Cobalt catalysts are considered the most effective for the synthesis of long-chain hydrocarbons. These catalysts are optimised for conversion of stochiometric syngas mixtures, mainly coming from the natural gas. On the other hand, iron-based FT catalysts are normally used for the conversion of synthesis gas from coal and they are promising catalysts for the conversion of biomass as they possess water–gas shift properties (i.e. altering the hydrogen to carbon monoxide ratio in synthesis gas). At the ESRF, users study both types of catalysts.

Scientists are trying to find out what happens during the FT reaction and with this knowledge they wish to design more active and stable catalyst materials. The deactivation of a FT catalyst results in a gradual decrease in the FT reaction rate. There are many possible reasons for the FT deactivation, such as reoxidation of active sites, sintering, catalyst poisoning from impurities in the syngas feed, polymeric surface carbon formation or surface metal-support compound formation (Tsakoumis *et al.* 2010). FT catalysts operate in multiphase medium and at higher pressures and temperatures. Not so long ago, the only way to study these catalyst systems was through *ex situ* methods of catalyst characterisation. These are not reliable due to the changes that the structure can undergo when withdrawn from the reactor. For example, cobalt metal particles can be reoxidised when exposed to air.

Using synchrotron methods and with a proper sample environment where the reaction conditions can be reproduced (i.e. high temperature, syngas reaction environment and preferably high pressures), scientists can now perform a direct *in situ* or operando characterisation of a catalyst in FT reactions. The wide array of techniques allows scientists to get information on different aspects of catalysis structure, such as the nature of crystalline phases, sizes of metal and oxide nanoparticles (X-ray diffraction) or the oxidation state of the atoms surrounding the catalyst (X-ray absorption fine structure spectroscopy or XAFS). Where possible, these synchrotron-based techniques are directly combined with more conventional methods, such as Raman spectroscopy.

At the Swiss Norwegian Beamline (SNBL – BM1), scientists have tested cobalt catalysts

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over several years. The team at SNBL have built an *in situ* cell and set up in collaboration with the Norwegian University of Science and Technology (NTNU) in Trondheim. In the cell, they include a small catalyst and a reaction mixture and observe the state of catalyst under industrial conditions. According to Olga Safonova, scientist at BM1: "This sample environment allows reproducing the experimental conditions (high pressure up to 20 bar, high temperature, space velocity of syngas, and FT reaction rates) close to the ones used in industry. The set-up is suitable to perform structural studies on working FT catalysts using X-ray diffraction, X-ray absorption spectroscopy, and Raman spectroscopy."

A team from the University of Lille recently carried out experiments on an alumina-supported cobalt catalyst using X-ray diffraction. The results showed that at a pressure of 20 bar and a temperature of 220°C, i.e. industrially relevant conditions, cobalt sintering took place during three to five hours of reaction. After eight hours of experiment, the cobalt would transform into cobalt carbide. These two processes provoked the deactivation of the FT reaction on their sample (Karaca *et al.* 2010).

In similar conditions, the team from the Norwegian University of Science and Technology carried out experiments using X-ray diffraction and X-ray absorption near edge structure (XANES) to reveal information about cobalt-based FT catalyst promoted with rhenium. Small amounts of noble metals like rhenium are added to FT synthesis in order to improve it. In the case of rhenium, it increases dispersion of the catalysis. This time the scientists studied the changes in the cobalt crystallites during FT synthesis in two different conditions: first, at FT synthesis conditions (483 K, 18 bar and low gas hourly space velocity) no significant changes in the cobalt crystallites was observed during the first hours of reaction. Cobalt carbide was not formed in these conditions. Running the reaction at higher temperatures and predominantly methanation conditions led to significant sintering of the cobalt particles and a further reduction of a partially reduced catalyst. This demonstrates how sensitive this complex reaction is to the employed reaction conditions (Ronning *et al.* 2009).

Iron-based catalysts have similar mechanisms to the cobalt ones. They consist of a precipitated iron oxide phase to which elements are added to improve the catalyst process. Scientists from the University of Utrecht (the Netherlands) went to the DUBBLE beamline (BM26) to measure the reaction process in different iron-based FT systems, including different amounts of additives. Typical so-called promoters are copper, potassium and silicon dioxide. The latter is added to disperse iron phases and to prevent sintering and attrition of the active catalyst, copper improves the reducibility of the catalyst and potassium is used to improve the selectivity of the catalyst towards longer hydrocarbon chains. The team combined XAFS and WAXS techniques to get complementary information. WAXS showed crystalline phases to be present after activation and during FT synthesis, and XAFS analysis suggested that for some catalysts, the majority of iron was present in amorphous phases, which were harder to detect by WAXS (de Smit *et al.* 2009).

Industrial use

This type of research might seem very fundamental, but FT technology is already being used in several pilot and large commercial refineries all over the world (South Africa, Malaysia, Qatar, China) and most of the work done at the ESRF is somehow linked to industrial uses: names like Shell, Total or Statoil often appear in the scientific results. The probable worldwide leader in FT research, though, is Sasol, a South African company that has been making energy this way since 1950. Sasol has made the most of the coal mines in South Africa and it extracts nearly 50 million tons of coal annually.

Researchers from SASOL have already visited ID31 twice trying to find the structure of a carbide generally considered responsible for the FT activity in iron-based FT systems. The second time, they brought with them a reaction chamber at 20 bar that could reach up to 300°C, in order to reproduce the exact conditions in industry.

M Capellas

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N E Tsakoumis *et al.* 2010 *Catalysis Today* **doi:10.1016/j.cattod.2010.02.077.**

TECHNICAL REPORTS

Energy Research at the ESRF

Edited by GARY ADMANS AND ANDY FITCH
European Synchrotron Radiation Facility, Grenoble, France

The modern world consumes ever increasing amounts of energy to sustain human numbers and contemporary lifestyles. Research and development of energy supplies and their exploitation are consequently topics of major economic, political and scientific importance. Moreover it is clear that current reserves of energy, predominantly fossil fuels, are limited, and there is widespread apprehension about the climatic effects of returning large amounts of carbon dioxide and other green-house gases to the atmosphere. Thus there is a degree of urgency to such studies. Energy systems are often of a complex nature. Development of new energy sources and improving the efficiency and exploitation of existing systems require detailed understanding of both structure and behaviour at the fundamental microscopic level, an area where the powerful X-ray beams of a synchrotron radiation source can play a major role. Thus energy research has become a theme of great scientific and practical importance at institutes such as the ESRF, which serves the scientific priorities of the research communities of its member countries.

In this report we present a few examples of recent research, a subset of the many other studies carried out on ESRF beamlines by users of the facility. Topics illustrated include nuclear energy, where X-rays help make current nuclear technology safer and cleaner, while preparing the groundwork for future generations of fusion reactors. Making the most of existing resources is another challenge. How do we make good use of the dwindling supplies of oil, and how best to convert sustainable biomass into fuel for transportation? Solar cell technology improves continuously. X-ray studies are used to characterize silicon wafers, to help optimize their manufacture and make them more reliable, and for fundamental studies of new solar-energy materials. Technologies based on hydrogen also show promise, but there are constraints to be overcome in storing and using this fuel. X-ray studies provide insight into the conditions at the center of a working fuel cell and help develop new components.

Imaging, diffraction, and spectroscopy allow detailed investigation of the structure of materials and their behavior while processed *in situ*. The variety of sample environments at beamlines is one key to the diversity of research. Specific reaction chambers mimicking industrial plants make possible *in operando* studies allowing scientific insight at the heart of a reaction process. These are some of the features of modern synchrotron research that enhance today's energy research and technology for a cleaner and more productive future.

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Fischer-Tropsch synthesis is a key step in the production of fuel or chemicals from carbon sources alternative to crude oil, such as gas, coal or biomass. Commercial plants employing this process typically convert natural gas to high-quality diesel. The reaction involves the catalytic conversion of syngas, a mixture of carbon monoxide and hydrogen, into long-chain hydrocarbons. The catalyst is usually iron or cobalt and reaction conditions involve high temperatures and pressures. Although well proven and used on a very large scale, the industrial process does have complications such as gradual deactivation of catalysts. Research at ESRF beamlines has provided insight into this reaction *in situ,* using reaction cells designed to mimic industrial reactors. At ESRF CRG beamline BM01 (SNBL), the structure–activity relations for alumina-supported cobalt catalysts were studied under realistic conditions using time-resolved X-ray diffraction and catalytic measurements [7]. Two processes for deactivation of the catalyst were witnessed, sintering of the cobalt and carbidization. Further information was gained in a study whereby XANES was used to confirm the oxidation state of the cobalt species under similar reaction conditions [8]. On ESRF CRG beamline BM26 (DUBBLE), another team of researchers has been investigating iron-based catalysts, which are promising for biomass conversion [9]. They used *in situ* XAFS and WAXS to look at the iron species present in the reaction for supported and unsupported catalysts. They also found suitable pre-treatment conditions for the catalyst that gave higher reactivity in the catalytic synthesis. These

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Hydrogen and gas storage

Hydrogen is a clean fuel alternative for transportation via combustion or generation of electrical energy in fuel cells. Under investigation at synchrotron sources are materials capable of storing this gas so that it can be used away from the site of production in a safe manner – not an easy task given the volatile nature of hydrogen gas.

Light-weight hydrides are seen as future hydrogen storage materials: they have high hydrogen capacity and some desorb hydrogen reversibly. Diffraction studies of structures and transformations lead to an understanding of the solid state chemistry and properties of such hydrides.

Studies based on pressure and temperature changes can reveal new polymorphs of a hydride and their structural evolution. X-ray powder diffraction, high-pressure techniques utilizing diamond anvil cells, DFT calculations and a symmetry-based analysis were used with ESRF CRG beamline BM01 (SNBL) to study the NH_3BH_3 and $M(BH_4)_n$ systems. Directional iono-covalent M . . . BH_4 and dihydrogen N-H . . . H-B bonding were revealed [15,16]. Understanding the structure makes it possible to design new hydrides with better thermodynamic stability. The temperature of hydrogen desorption could be reduced to a more favorable level (80–100°C) in borohydrides containing a combination of alkali and transition metals such as the Zn-based borohydrides, e.g. $MZn_2(BH_4)_5$ (M = Li, Na) [17] (see Figure 6). Other chemical derivatives were

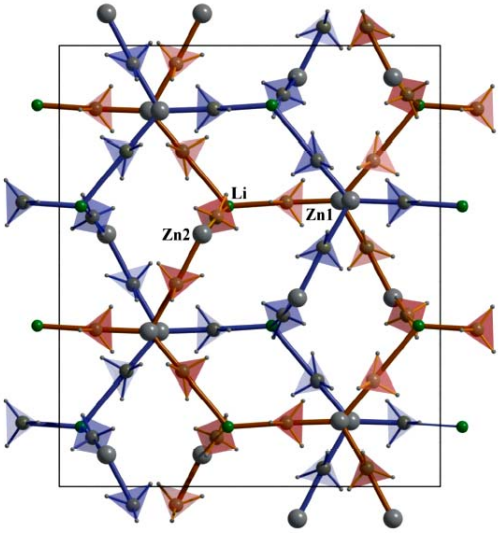

Figure 6: Crystal structure of $LiZn_2(BH_4)_5$. The doubly interpenetrating 3D framework is shown in blue and red (image courtesy of Y. Filinchuk [17]).

prepared by solid-state synthesis and their stability and decomposition reaction pathways studied by *in situ* powder diffraction. The most recent novel compositions included bimetallic $MSc(BH_4)_4$ (M = Li, Na, K) [18], $Li_4Al_3(BH_4)_{13}$, transition metal borohydrides $Y(BH_4)_3$, $Mn(BH_4)_2$, and mixed-anion $Li(BH_4,Cl)$ and $KZn(BH_4)Cl_2$.

The optimization of materials under realistic conditions was also investigated using *in situ* powder diffraction. The kinetics and thermodynamics of parallel reactions, hydrogen cycle stability of various Mg-based systems, including Mg-La [19], Mg-Fe-Co [20] and Mg-V, have been studied. The reversibility of borohydride-based composites with high hydrogen content has also been demonstrated.

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SNBL-ESRF workshop on diffuse scattering in crystalline materials

As part of our policy of seeking ways to facilitate the exchange of information between the SNBL team and the synchrotron user community, the SNBL management has organized a series of workshops at the ESRF based on themes of current and future interest both to the SN community and a wider scientific audience. The first of these meetings on the topic of nanoscale materials was held in 2006, followed by a workshop on the subject of high gas pressures for *in-situ* experiments in 2007. Both of these meetings were attended by 30-40 participants, including participants from various synchrotron beamlines as well as external users. The next meeting, which took place in June 2008, continued the theme of *in-situ* experiments, but specifically includes the use of our new Raman spectrometer in combination with x-ray absorption and diffraction measurements. 80 participants, came from 14 countries.

The first joint SNBL-ESRF workshop took place June 25-26 2009 at the ESRF, Grenoble. The goal of this 2-day workshop was to bring together people interested in diffuse scattering, in order to share their experience in experiment and theory and to present and discuss new problems in physics and chemistry in which disorder phenomena are important.
More than fifty participants from 10 countries (France, Switzerland, Norway, Germany, Austria, Russia, Spain, Australia, UK, USA) created a very stimulating, constrictive and friendly environment for exchanging theoretical ideas and experimental know-how. The list of participants shows the presence of world-leading experts in the field of diffuse scattering such as Hans-Beat Bürgi (Switzerland), Richard Welberry (Australia), Tai-Chang Chiang (USA), Reinhard Neder (Germany), Harold Reichert (Germany/ESRF), Sergey Wakhrushev (Russia), together with young scientists and Ph.D. students. The study of disordered phenomena has a long tradition in Switzeland and Norway; there were 10 Swiss and 3 Norwegian participants. There were also visitors from ESRF and ILL interested in diffuse

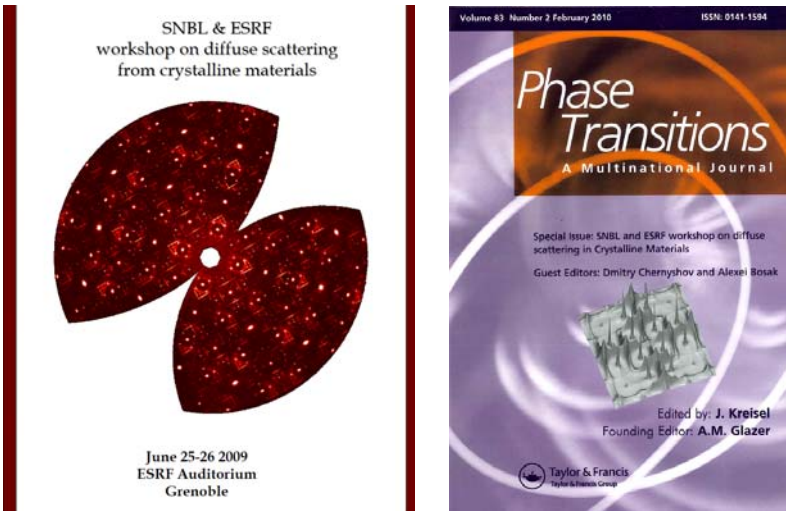


Figure 1. Programme booklet and Proceedings of the Workshop

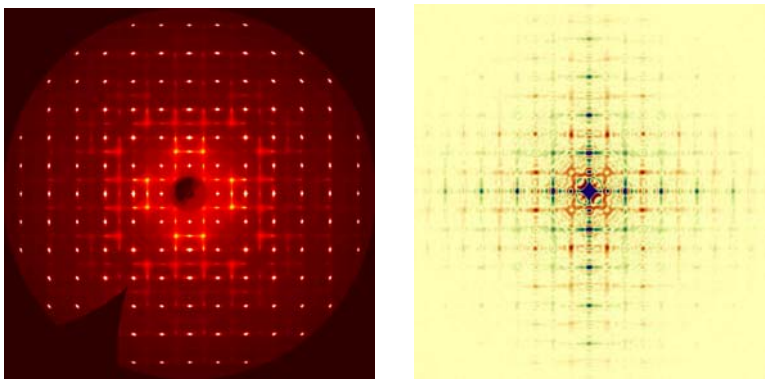


Figure 2. LEFT: A diffuse scattering map measured at SNBL (Chernyshov, D. and Bosak, A.(2010) 'Diffuse scattering and correlated disorder in manganese analogue of Prussian blue', Phase Transitions, 83: 2, 115 — 122). RIGHT: corresponding autocorrelation function in XY0 section, calculated with help of a new analysing tool developed at ETHZ (courtesy of A. Simonov and T. Weber).

scattering.
There were many interesting lectures presenting new approaches in data analysis, illustrating the use of combination of diffuse and inelastic scattering techniques, showing new challenging problems to be solved in future, and offering new experimental methods. To have a record of the main results, a selected collection of presentation was published in a special issue of "Phase Transitions" international journal.

The workshop also gave an opportunity for our users to see what can already be done at ESRF and SNBL today and, with their help, what might be possible in the future. For a synchrotron facility offering beamtime for user community, such as SNBL or ESRF beamlines, the workshop helped to define the direction in which we have to develop in order to fulfil the requirements of our user community. In particular, it becomes clear that the best data quality of diffuse scattering experiments can be achieved with a PILATUS detector; and a corresponding proposal for detector and goniostat has been therefore submitted. We are offering now 3D inspection of reciprocal space that can be done shortly after data collections with help of software developed together with colleagues from ID28 ESRF beam line. Fast and reliable measurements of diffuse scattering data assume that there will be a large amount of information to be analysed and require fast computers operating with large amounts of graphical information. Analytical tools facilitating data analysis have been in particular developed at ETHZ and tested with data collected at SNBL.

The workshop also gave an example of constructive collaboration between SNBL and ESRF in terms of organization and finance issues. Scientific collaboration with ID28 has been successfully continued and we have 7 common publications, 4 more are submitted. This fruitful combination of mapping of diffuse scattering with inelastic scattering could be further explored by Swiss and Norwegian users as well as our know-how and experimental protocols on data collection and analysis.

STATUS OF FACILITY

BM01A

The period 2009-2010 has seen an unprecedented increase in the productivity of the beamline, particularly in the number of high profile publications (see the full list attached to this report). In comparison with earlier years, there has been a noticeable shift towards more physics-oriented journals (***Phys.Rev.Lett.*** and ***Phys.Rev.B***) rather than typical high impact chemical journals such as ***Angew. Chemie*** or ***JACS***. The reasons behind this trend are manifold. On the one hand, we have been able to offer new equipment which permits our users to probe more extreme conditions e.g. high pressure cells or low temperature cryostats. Experiments of this type tend to attract users interested in the fundament physical properties of the system under investigation, particularly those scientists coming from the neutron scattering community. This has led to a very fruitful collaboration between the staff of SNBL and users coming from the Laboratory for Neutron Scattering at the Paul-Scherrer Institute, resulting in several excellent publications. In addition, the SNBL-ESRF Workshop on Diffuse Scattering which took place in 2009 served as a springboard for a number of proposals involving ESRF beamline staff in collaboration with SNBL. The combination of results from X-ray elastic, inelastic and diffuse scattering has provided material for a number of papers which have already appeared in print, and several more which are under review. The data for some of these publications have been collected on several different beamlines, including data from a large area Pilatus6M detector at the Swiss-Light-Source. This trend is likely to continue, at least until our own pixel detector will be available at SNBL.

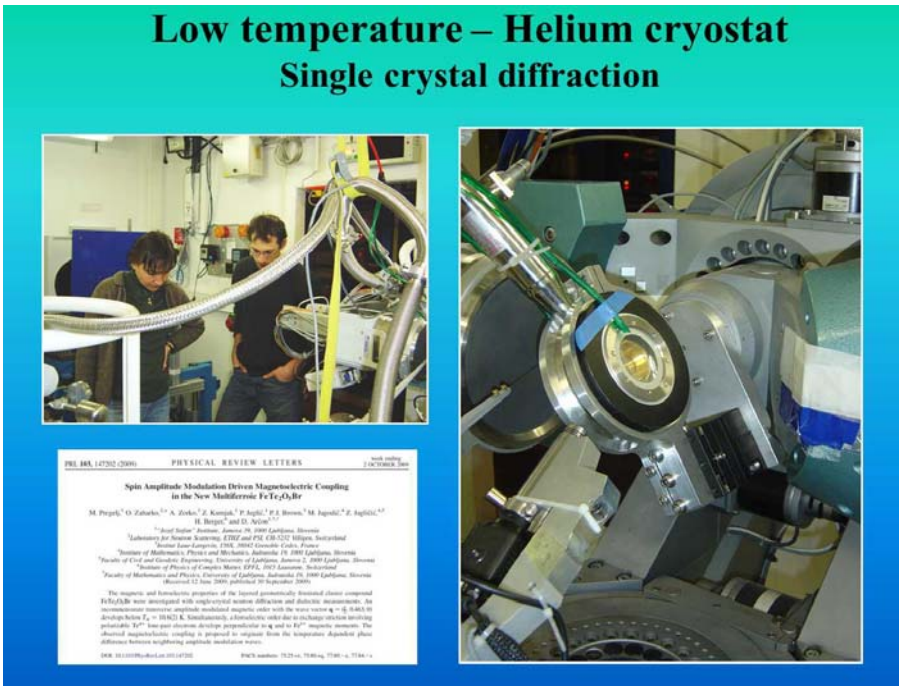
The SNBL project has benefited from a very generous equipment loan from Prof Walter Steurer, Head of the Laboratory of Crystallography of the ETH Zurich. We are pleased to acknowledge that Prof Steurer has given SNBL (on long-term loan) a wide range of high pressure equipment, including several Diamond Anvil Cells, replacement diamonds and a considerable amount of ancillary equipment. The availability of such an excellent selection of high pressure equipment at SNBL has transformed both the quality and quantity of experiments which can be carried out in

this regime of extreme conditions, and there has been a corresponding jump in the number of publications from SNBL in the field of high pressure research.

The opportunities for collecting high quality diffraction data from single crystals in the temperature regime down to a few degrees Kelvin are very limited indeed, even at synchrotron radiation facilities. We have purchased and commissioned a miniature cryostat which was originally designed for low temperature optical microscopy. With a change of window material and minor mechanical modifications to the housing, we can now operate this cryostat down to a base temperature of 5 Kelvin either on the mar345 image plate or the KM6 multi-axis diffractometer. The first paper which appeared using this cryostat concerned the investigation of a new multiferroic material $FeTe_2O_5Br$ by a group from PSI, published in Phys. Rev. Lett. in 2009. Several groups have expressed interest in using this device, and we will shortly be carrying out experiments using laser photo-excitation at 10 K (University of Geneva, Prof. A Hauser).

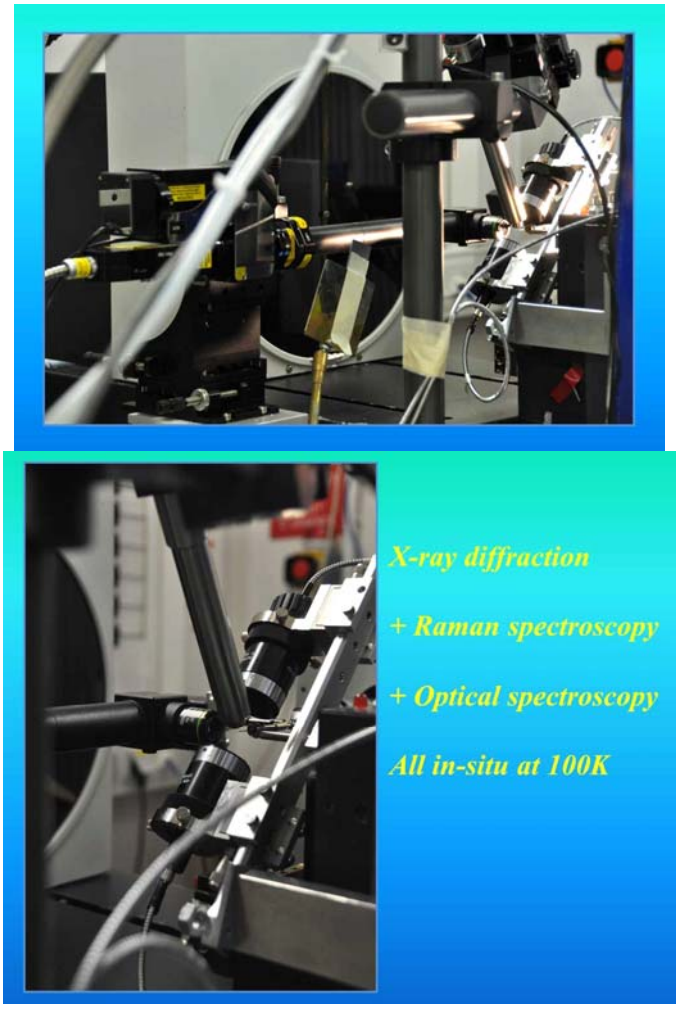
The combination of experimental techniques for in-situ experiments continues to be a popular theme at SNBL. The use of a Raman spectrometer in conjunction with X-ray diffraction measurements has, in the meantime, become a standard technique on BM01A. Recently, we developed this idea somewhat further, and a group from Oslo (Kristoffer Andersson, University of Oslo) managed to carry out both Raman and optical spectroscopy on samples at 100K, during a diffraction experiment. The set-up around the sample became somewhat crowded, but in the end we achieved a configuration which is probably unique in synchrotron experiments!

Another example of the use of a combination of techniques concerned the in-situ synthesis and crystallization of a Zr-Metal-Organic-Framework structure using powder diffraction and Raman. This was the first time that an in-situ crystallization

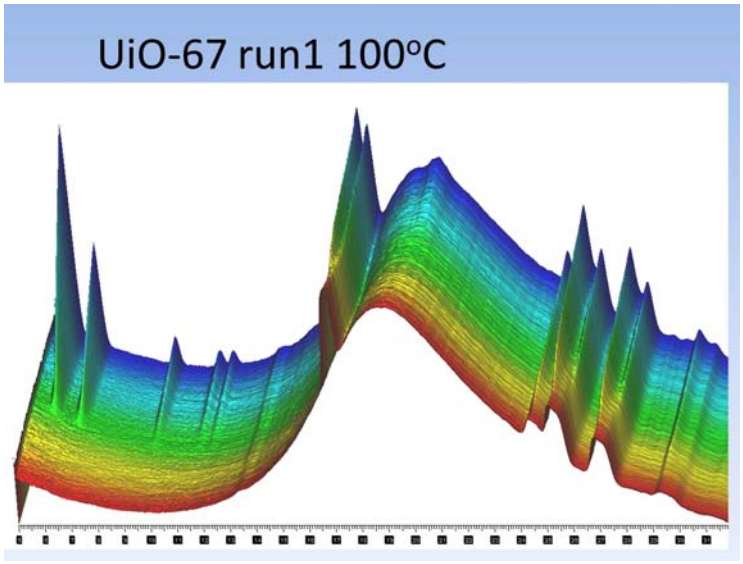


experiment had been attempted at BM01A, and the results were of very encouraging. An extract from one of the the powder diffraction patterns is shown here, illustrating both the quantity and quality of the data (Karl-Petter Lillerud, University of Oslo)

All time-resolved experiments are ultimately limited by the read-out cycle of the detector system. In order to improve the time resolution in our in-situ experiments, and, of course, to achieve a higher through-put for all diffraction experiments, we have proposed the purchase of a new CCD detector for the KM6 diffractometer and a replacement pixel detector for our ageing image plate reader. These suggestions are in line with the priorities for SNBL which were identified by the Committee of the Future and form part of their main recommendations. At the time or writing this report, a purchase order for a new CCD detector has been placed with Oxford Diffraction (now part of Agilent Technologies) with delivery expected in March 2011.
A design study has also been carried out for replacing the image plate with a Pilatus2M pixel detector, and we are awaiting confirmation of the funding. With the aid of these detectors of the very latest generation, SNBL can continue to provide a modern diffraction platform for our user community.

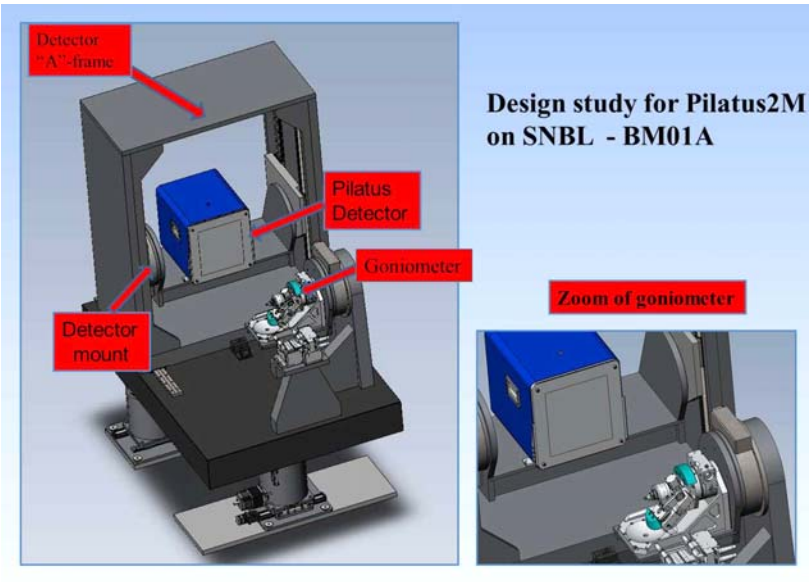


Photos of the set-up for combined spectroscopy and diffraction experiments on the mar345 image plate diffractometer.



Powder diffraction patterns as a function of time, showing the onset of crystallization.

In addition to the technical improvements outline above, we have also continued to develop our contacts with other synchrotron beamlines and facilities. In addition to the long-standing collaboration agreement with the Dutch-Belgian beamline (DUBBLE) at the ESRF, we now have a Memorandum of Understanding (MoU) with MaxLab. The goal of this new MoU is to promote the exchange of knowledge and experience with the operation of diffraction equipment on synchrotron beamlines. We have already hosted two scientists from MaxLab (Jeppe Christensen and Dörthe Haase), and we have begun a series of round-robin measurements in which the same sample will be measured at difference facilities. This will allow us to establish the strengths and weaknesses of each facility, which will be of mutual benefit to all participants. A part of these measurements has already been carried out at BM01A, and similar data sets will be collected shortly on the new single crystal diffractometer being installed at MaxLab and at the Diamond synchrotron in England.



BM01B

Originally the B station of the Swiss-Norwegian beamlines (SNBL, BM01B) at the European Synchrotron Radiation Facility (ESRF, Grenoble, France) was designed to perform ex-situ high resolution powder diffraction (HRPD) and ex-situ X-ray absorption fine structure (XAFS) experiments. During the last decade the station has been transformed into a dedicated beamline for combined *in-situ* X-ray diffraction and X-ray absorption experiments. Practically the complete station has been redesigned, reorganized and rebuilt and step by step commissioned while maintaining a complete user program. Not one single original piece of the optical X-ray elements has survived during the transformation. The beamline controls and software are also all brought to modern standards. As a result, the station is now equipped with two monochromators: a HRPD-dedicated channel-cut monochromator and a second double crystal monochromator for XAFS (Fig.1).
Both monochromators can be easily and rapidly (within seconds) interchanged. Besides these two traditional synchrotron-based techniques the experiments can also be followed by Raman scattering. The Raman spectrometer is located outside the experimental station. The exciting laser light as well as the Raman scattered signal are transferred to and from the sample with optical fibres of ~50 m length. The station is now capable of performing quasi-simultaneous HRPD, XAFS and Raman scattering studies on dynamic systems allowing to elucidate many different aspects of complex reactions at the same time on the same sample spot.

Combined studies are hence often done as a function of some externally varying parameter. The SNBL has a pool of equipment filled with a wide variety of 'in-situ instruments', which are either custom-designed or adapted to the beamline. As an example a capillary flow cell or microreactor is shown in Fig.2.
The SNBL offers this equipment to experimentalists as a part of its user service. Some of these instruments are relatively standard: Temperature control via a hot-air blower, a cryo-stream and a cryostat offering a temperature range from 4.5 to 1200 K. However, more parameters have to be accessed when performing so-called operando studies. During operando experiments scientists try to reproduce the realistic conditions of a chemical process (e.g. composition of reactive media, pressure, space velocity of reactants etc.) at the beamline (or in the laboratory) while following the structural changes of the working catalyst or other functional materials such as gas sensors or electrochemical devices. Combined HRPD, XAFS

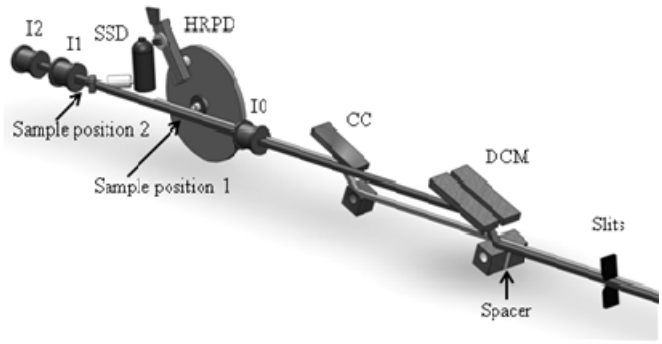


Figure 1. SNBL-B station schematic layout

and Raman data taken during various stages of a chemical process enable a better understanding of structure/activity/function relationships. The success of such multi-technique approach relies upon the rigorous optimisation of many experimental details. During the last 2 years the B-station performance has reached a very high level of integration. Experiments which used to be extremely complex and challenging have now become routine.

Future developments:

Thanks to the success of the unique combined approach and enthusiastic user feedback, we are inspired to continue our developments. Now that we have reached a stable and high performance level of all installed equipment, we have started to prepare the future improvements. All our plans have one major component in common *"time"*. We aim at pushing the *time resolution limits* of our machines in three ways:

- Increase photon flux density
- Implementation of new 2D detectors
- Improved sensitivity and selectivity though modulation enhancement.

A design study has been made to check the feasibility of installing collimating pre-mono mirrors and post mono focussing mirrors (Fig.3). For this project we have recently obtained the go-ahead from our management. These changes consist of major design, technical and logistical efforts. The first vacuum hardware preparations and installations are planned in the coming 2 years.

The purpose of these mirrors is many-fold:
A) Gain in intensity: The collimating first mirror should allow to accept more vertical beam without spoiling the resolution of the following monochromator.
B) Harmonic rejection will ensure true monochromaticity of the beam without detuning of the monochromator. This will in itself already lead to a higher flux.
C) Reduction of the heat load (and the resulting distortions and intensity losses) on the first monochromator crystal.
D) The suppression of high energies in the incoming beam will also boost the lifetime of monochromator components (seals, cables, windows etc) thus increasing the overall reliability of the beamline.

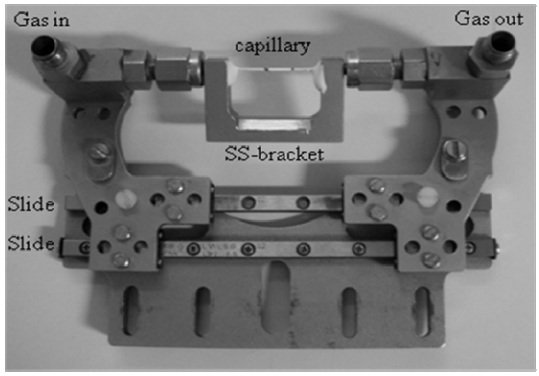


Figure 2. Custom designed capillary flow cell

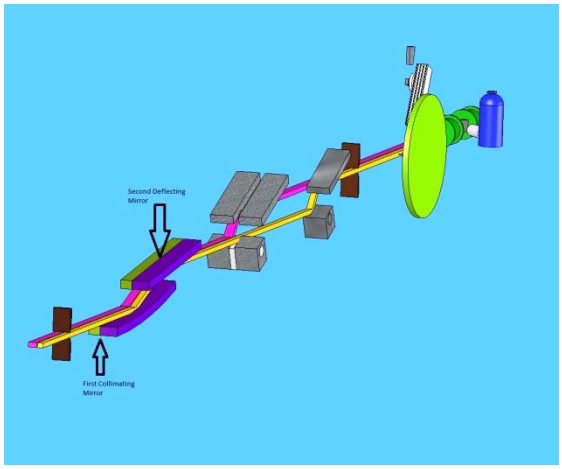


Figure 3. Design study beamline layout including 2 pre-mono mirrors

For powder diffraction we have already installed the latest generation pixel detectors (Mythen) capable of collecting a full pattern in scanning mode. Optimization and calibration is ongoing. This detector will bring at least one order of magnitude in signal-to-noise.

Chemical and physical processes of technological or fundamental importance are often rather complex. The need of understanding and controlling their function and performance is our main driving force for developing new experimental techniques. We have already tested a further step towards sensitivity and selectivity amplification of the experiments based on synchrotron radiation: Modulation Enhanced Diffraction (MED) and Modulation Enhanced Spectroscopy (MES). These novel techniques utilize the periodic variation of an external parameter, such as temperature, concentration, pressure, light flux and electric field, to influence in a periodic way the properties of the responding moieties of interest (i.e. the active ones) in the sample, which can go from the atomic or molecular level to mesoscopic

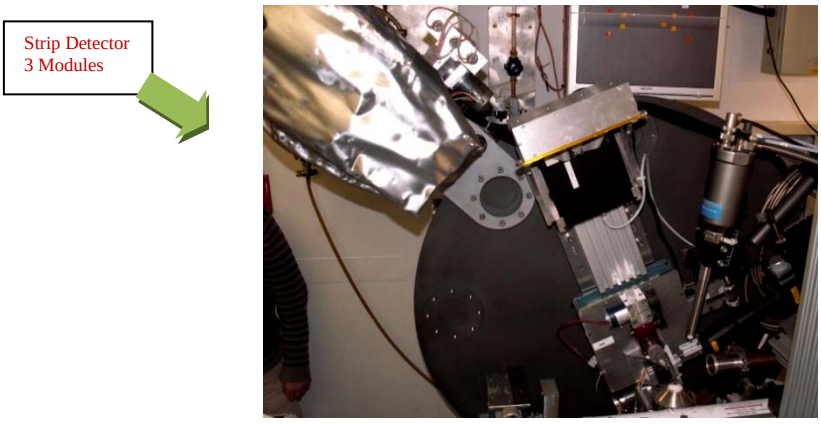
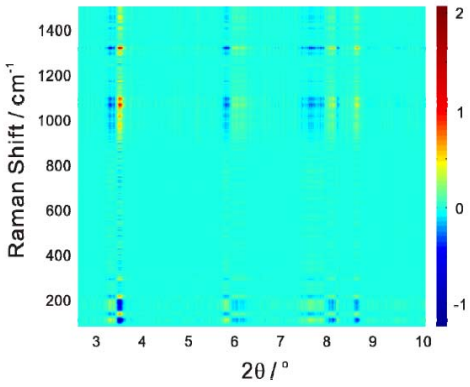


Figure 4. Mythen Strip Detector, freely rotatable inside the 6-analyzer crystal array

nano-arrays up to bulk crystalline phases. The periodic response appears in the measured intensity and can be sensitively and selectively detected by the post-experimental mathematical treatment. We intend to further develop the modulation technique that would cover X-ray powder and single crystal diffraction (MED), Raman spectroscopy (Raman-MES), and X-ray absorption spectroscopy (XA-MES), together with the multidimensional correlation analysis of the data.



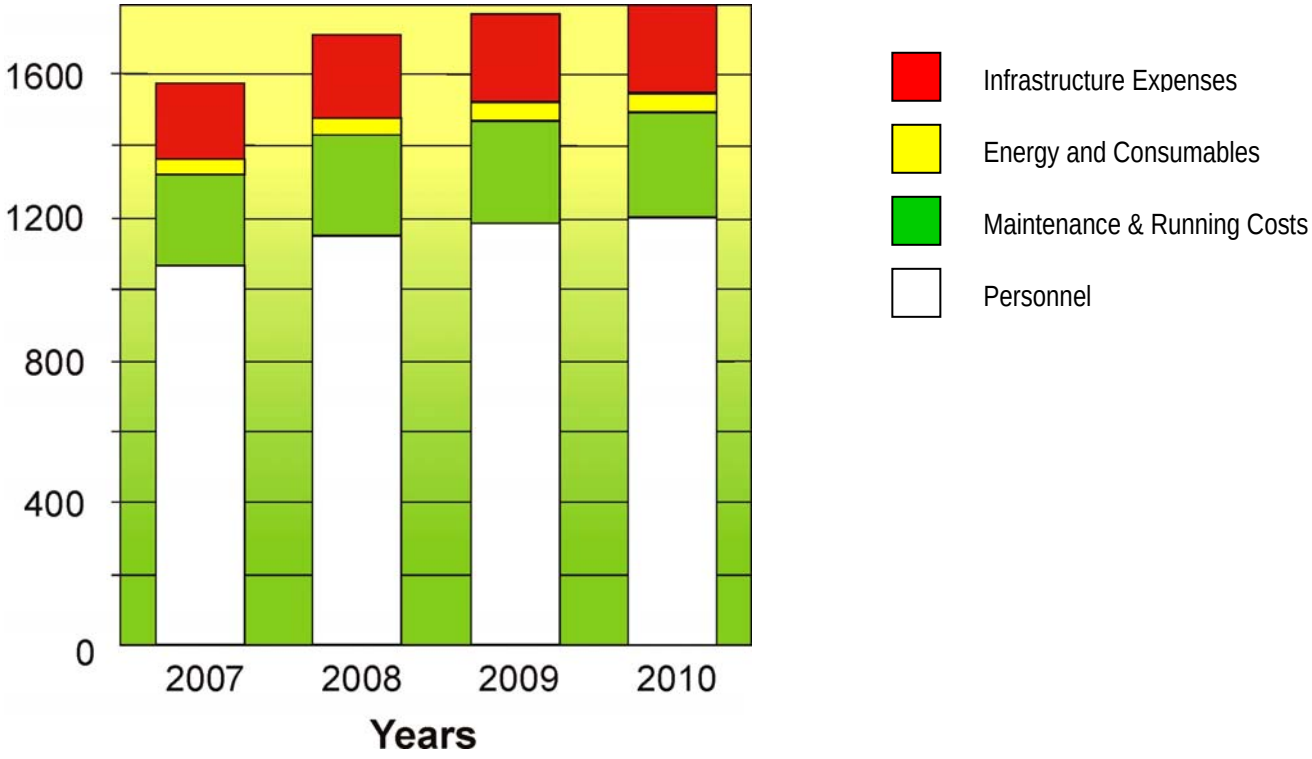
$$C(2\theta, \nu) = \int_0^T A(2\theta, t) \cdot B(\nu, t)dt \qquad C(2\theta, \nu) = \int_0^T A(2\theta, t) \cdot B(\nu, t)dt$$

Figure 5. Multidimensional correlation analysis Raman-XRPD

Reproduced from Urakawa, A. et al. *Combined, Modulation Enhanced X-ray Powder Diffraction and Raman Spectroscopic Study of Structural Transitions in the Spin Crossover Material [Fe(Htrz)$_2$(trz)](BF$_4$)* J. Phys. Chem. C, 115, 4, 1323–1329, 2011

SNBL - FACTS AND FIGURES

BUDGET (in kCHF)

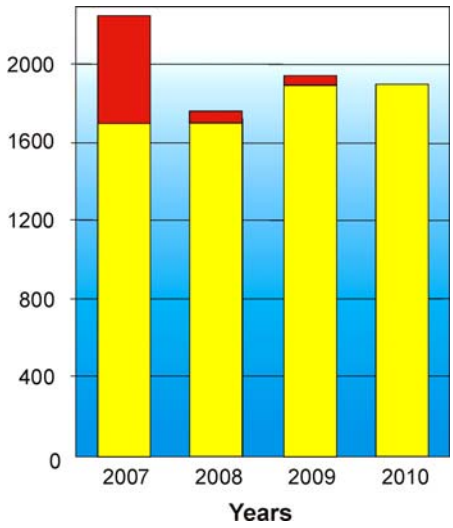


BUDGET in kCHF	2007	2008	2009	2010
Personnel	1,064	1,144	1,174	1,200
Maintenance and Running Costs	257	275	280	290
Energy and Consumables	45	50	52	53
Infrastructure Expenses	210	236	244	247
TOTAL	**1,576**	**1,705**	**1,750**	**1,790**

INCOME (in kCHF)

Income Outside Contract

Income According to Contract



INCOME in kCHF	2007	2008	2009	2010
Income According to Contract	1,698	1,663	1,750	1,790
Income Outside Contract	543	54	66	-
TOTAL	**2,241**	**1,717**	**1,816**	**1,790**

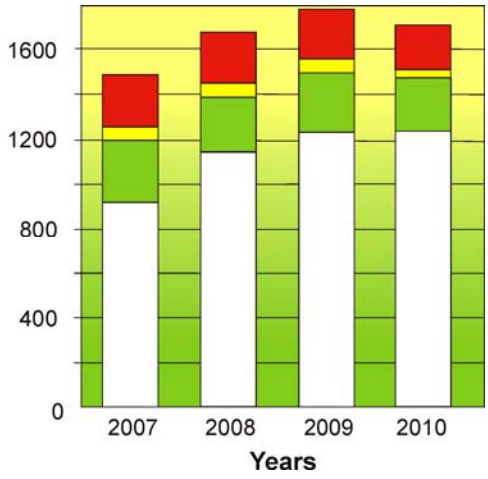
EXPENDITURE (in kCHF)

Infrastructure Expenses

Energy and Consumables

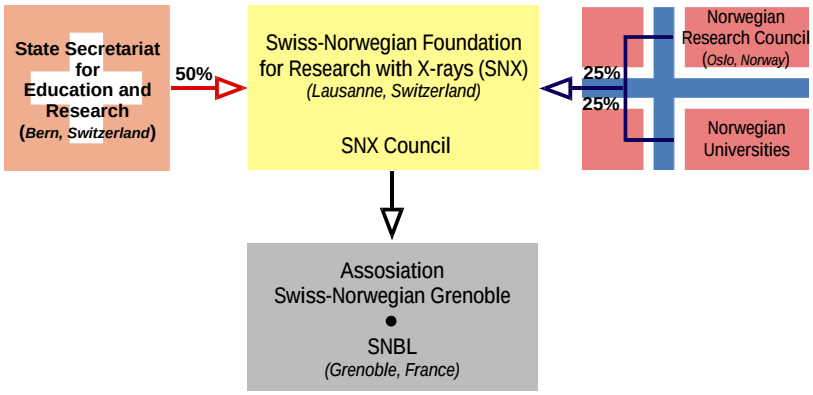
Maintenance & Running Costs

Personnel



EXPENDITURE in kCHF	2007	2008	2009	2010
Personnel	1,030	1,144	1,238	1,243
Maintenance and Running Costs	342	244	262	234
Energy and Consumables	56	62	65	41
Infrastructure Expenses	240	227	220	199
TOTAL	**1,668**	**1,677**	**1,785**	**1,717**

Organization Chart of the SNBL



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(2009-2010)

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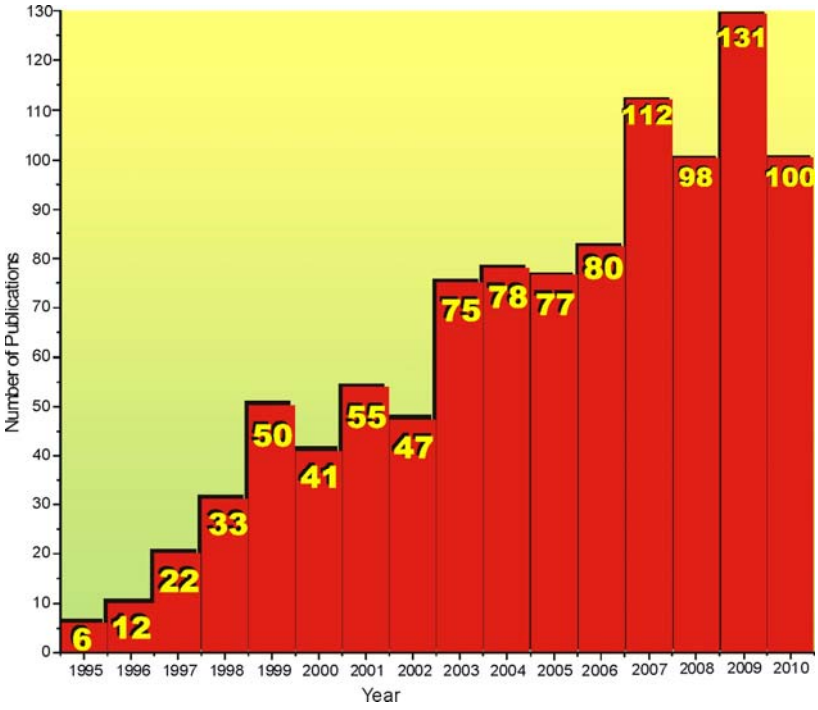
Dr. D. Chernyshov – BL scientist	**W. van Beek** – BL scientist
Dr. Y. Filinchuk – BL scientist	**Dr. O.Safonova** – BL scientist

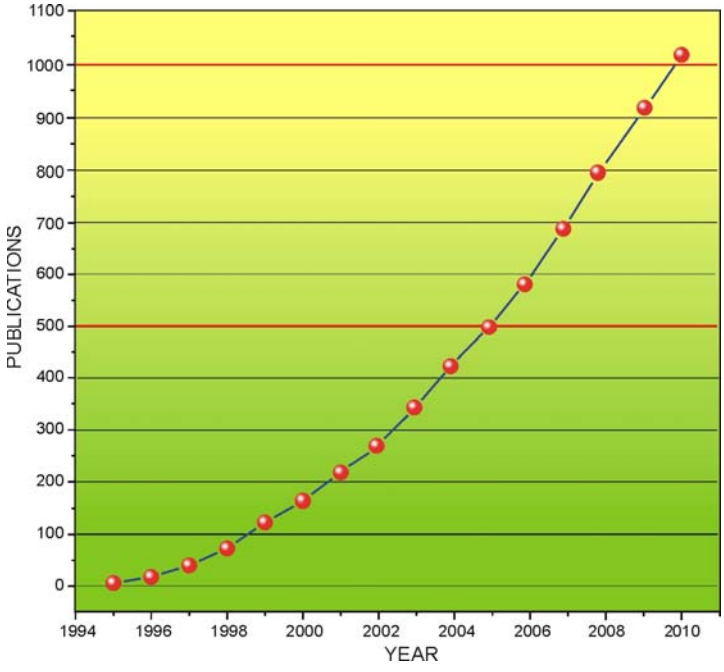
Ch. Heurtebise – Administrative manager

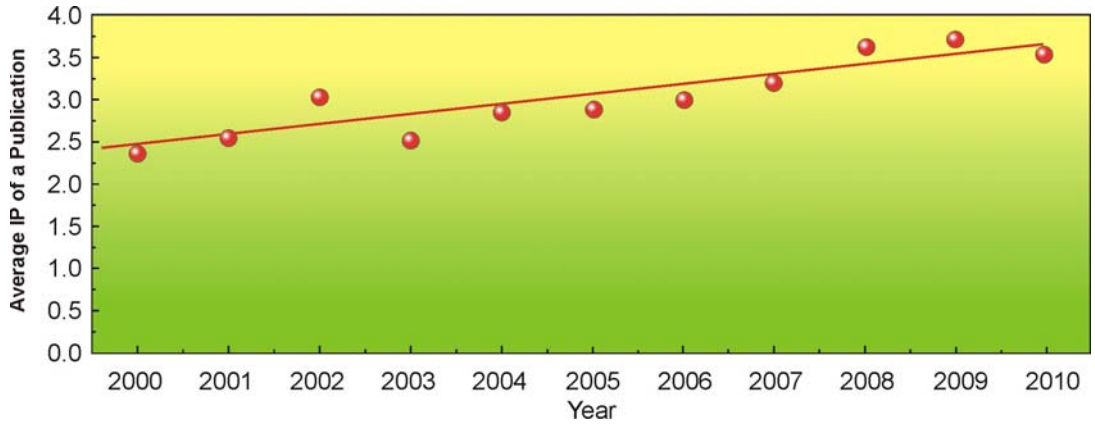
G. Wiker – Senior technician

PUBLICATIONS

Publication Rate since start-up of SNBL







Impact factor of the "average journal" paper produced by the SNBL Users. Straight line is the best least-square fit.

List of Publications

2009

1. **Afanasiev, P., Bouchu, D., Kudrik, E., Millet, J.-M., Sorokin, A**. *Stable N-bridged diiron (IV) phthalocyanine cation radical complexes: synthesis and properties* Dalton Trans., 9828 - 9836, 2009
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5. **Boldyreva, E.** *Combined X-ray diffraction and Raman spectroscopy studies of phase transitions in crystalline amino acids at low temperatures and high pressures: selected examples* Phase Transitions A, **82**, 4, 303 – 321, 2009
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10. **Calamiotou, M., Gantis, A., Lampakis, D., Siranidi, E., Liarokapis, E., Margiolaki, I., Conder, K.** *Pressure-induced phase separation in the Y123 superconductor* EPL, **85**, 26004-26010, 2009

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12. **Chambrier, M.-H., Le Bail, A., Kodjikian, S., Suard, E., Goutenoire, F.** *Structure Determination of $La_{18}W_{10}O_{57}$* Inorg. Chem., **48**, 14, 6566–6572, 2009
13. **Chen, W., Williams, A.J., Ortega-San-Martina, L., Li, M., Sinclair, D.C. et al.** *Robust Antiferromagnetism and Structural Disorder in $Bi_xCa_{1-x}FeO_3$ Perovskites* Chem. Mater., **21**, 10, 2085–2093, 2009
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29. **Førde, T., Næss, E., Yartys, V.A.** *Modelling and experimental results of heat transfer in a metal hydride store during hydrogen charge and discharge* Int. J. Hydrogen Energy, **34**, 12, 5121-5130, 2009
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