Schweizerische Gesellschaft für Kristallographie
Société Suisse de Cristallographie
Swiss Society for Crystallography

Sektion für Kristallwachstum und Kristalltechnologie
Section de Croissance et Technologie des Cristaux

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SGK/SSCr NEWSLETTER

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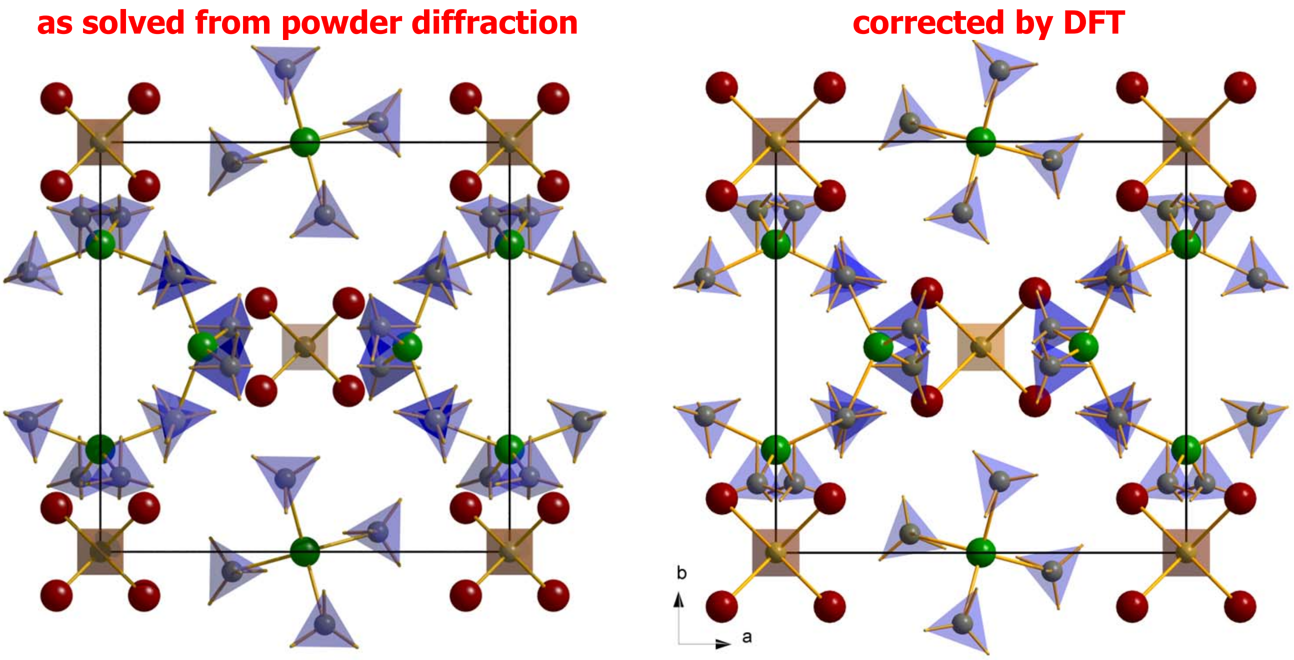


$Al_3Li_4(BH_4)_{13}$

In this issue: Program and abstracts of the annual Meeting, Sept. 16/17 in Geneva
Agenda of the general assembly 2010

On the Cover:

Structure of the novel Li-Al-borohydride complex $Al_3Li_4(BH_4)_{13}$ as solved from X-ray powder diffraction data (left), and corrected by the density functional theory (DFT) optimization (right). Li-Al-borohydride is a potential on-board hydrogen storage material since it shows decomposition at about 70°C combined with a high gravimetric (17.2 wt%) and volumetric (117 kg/m^3) hydrogen density.

(Chem. Eur. J., 2010, ***16***, 8707, see also abstract P20 on page 40 of this newsletter)

SGK/SSCr Newsletter No. 81

Flyers

Dear members of the Swiss crystallographic society,

Why are collaborations so crucial in science?

Let us have a look at the fundamental universal constants, which we use more or less every day in our science? Are they really correct? Only recently, the dramatic news that the proton was not the size (0.8768 fm), which we admitted for 60 years now… but it is apparently quite smaller [1].
How was this shown? To say it simply, take a hydrogen atom, consisting of a nucleus and an electron. The electron is such a light-weight that it is difficult to "look at". Now replace the electron by a negatively charged muon, which is ca. 200 times heavier than an electron and now compare… What sounds so simple was done in a ca. 10 years' project, and in a collaborative effort with many scientists of different institutions. The results for the size of a proton give a value of 0.8418 fm, about 4% smaller than the old value. What does this mean? If it proves correct, it means that something fundamental is wrong in particle physics. It is possible that the Rydberg constant R hasn't been correctly measured. This value describes the way light gets emitted from various elements and plays a key role e.g. in spectroscopy. Or, if the Rydberg constant is correct, the smaller size of a proton could mean that the equations in quantum electrodynamics theory will fail to work. Theories will have to be expanded, extended, and will certainly lead to more understanding on one hand, and many more questions and new problems on the other – altogether an advancement of knowledge in any case.
The development of new techniques to investigate such phenomena is of course crucial to such investigations, just about as much as the idea itself. Complexity of science, be it in physics, chemistry or biology, and in all fields in between, requires today a team of scientists working together to solve a problem. This is, in parallel to the problem itself, what makes our research so valuable.

With this, I wish you a happy research and a lot of results,

Katharina Fromm

[1] The size of the proton**.** *Nature*, 2010; 466 (7303): 213 DOI: 10.1038/nature09250

The International Summer School on "Diffraction at the Nanoscale: Nanocrystals, Defective & Amorphous Materials"

Contributed by Fabia Gozzo (Paul Scherrer Institute, PSI), Antonella Guagliardi (Italian Crystallographic Association, AIC), Radovan Černý (University of Geneva)

Workshop supported by the European Society for Crystallography

The Powder Diffraction School (http://user.web.psi.ch/powder2010/) jointly organized in Switzerland by the Italian (AIC) and Swiss (SSCr) Crystallographic Societies and by the Paul Scherrer Institute (PSI) on 24–30 May 2010 was focusing on a new, frontier, topic in crystallography and material sciences, such as the development of characterization techniques for the investigation of disordered, nanosized and defective material, for which the traditional Bragg approach fails.

The dense School program included lectures, demo sessions, tutorials and diffraction measurements at the Materials Science beamline of the synchrotron at the Swiss Light Source of the PSI. Lectures were typically given in the morning sessions with the entire audience in the main lecture room, while (plenary) demo session followed in the afternoons; two full days were allotted for tutorials (experimental sessions and data analyses) to students subdivided in smaller groups.
In the first day, Dr.s van Petegem and Malavasi and Dr. Černý and Domínguez-Vera introduced the vast realm of nanosized matter in Materials Science, Chemistry, Physics and Biology. In the following day, Prof. Scardi and Dr.s Gozzo and Zaharko presented the theoretical and experimental background necessary for a meaningful interpretation of the (x-ray and neutron) diffraction results of nanocrystals and nanoscale-ordered matter. Later in the week, Prof.s Billinge, Neder, Keen, Scardi and Dr.s Cervellino, Guagliardi, Leoni, Božin and Tucker presented the different approaches (Pair Distrubution Function, Debye Function, Reverse Monte Carlo, Whole Pattern Modeling, etc.), validated by the extensive use of the PDFGETX/N, DEBUSSY, WPPM, DISCUS, GUDRUN, DIFFAX+ suites of programs, on which the attendees got acquainted in the tutorial sessions. Finally, Dr.s Abolhassani, Diaz, Giannini and Favre-Nicolin introduced transmission electron microscopy analyses and the basic concepts of coherent diffraction imaging, including a number of very interesting applications in nanosized crystals and aperiodic objects.
In addition, the volunteering by all speakers made it possible to assemble a 220 pages book entitled "*Diffraction at the Nanoscale: Nanocrystals, Defective & Amorphous Materials*", edited by A. Guagliardi and N. Masciocchi. This book is specifically targeted to the new generation of crystallographers and material scientists, dealing with nanocrystalline and defective materials. Both the novice and the experienced scientists will likely find therein new and relevant scientific aspects, as well as a large bibliographic section. This book was freely distributed to all participants and is now available for a nominal price (15 EUR) from the Publisher, Insubria University Press (please contact Dr. Sara Visconti at iup@uninsubria.it).

The International Summer School Social Dinner took place in the heart of the cozy village of Villigen on Thursday evening, from which the group photo shown here is taken.
The lunches and dinners have been organized to encourage the participants to further discuss among themselves and with the teachers. Accordingly, students, teachers, and tutors fruitfully exchanged their ideas, skills, and experience throughout the entire week. Needless to say, it has been a very successful scientific week.

Further material for upload available at http://user.web.psi.ch/powder2010/.

News for and from members

We welcome the following new members of the SGK/SSCr:

Personal members

Dr. Antonio Cervellino
Paul Scherrer Insitut Swiss Light Source,
WLGA-229, 5232 Villigen PSI

Dr. Arthur Braun
EMPA, High Performance Ceramics, Überlandstr. 129,
CH-8600 Dübendorf

Abita Shyorotro Chimpri
University of Berne, Department of Chemistry and Biochemistry, Freiestrasse 3,
3012 Bern (PhD. student)

Travel grants for young SGK/SSCr members

The committee will award the grants according to the following rules:

- Preference is given to PhD students
- Proof has to be given that there are no grants available covering the expenses
- A supporting letter by the supervisor of the applicant is necessary

If you wish to apply for a travel grant, please send the above mentioned documents to the president of the SGK/SSCr anytime. You should have been member for at least one year before applying for a grant.

Travel grants are good opportunity allowing young scientist to profit from our society in a period with low income and returning this goodwill later as senior scientists being long-term member of our society to the next generation.

Details for applications are given at:

http://www.sgk-sscr.ch/TravelGrants.pdf

3rd Erwin Felix Bertaut Prize

The Third Erwin Felix Bertaut Prize of the European Crystallographic Association (ECA) and European Neutron Scattering Association (ENSA) is awarded to Dr. Tom Fennell, beamline scientist from the Institut Laue-Langevin, Grenoble, France.
Tom Fennell is recognised for the outstanding level of the neutron scattering experiments, using both polarized and high resolution techniques, in his pioneering work to characterize spin ice materials and demonstrate the existence of a Coulomb phase therein. His diffuse neutron scattering measurements in applied magnetic field or with polarization analysis have revealed the first examples of pinch point scattering in magnetic systems, underpinning theories in which the excitations of a spin ice are emergent magnetic monopoles.

5th Max Perutz Prize

The fifth Max Perutz Prize of the European Crystallographic Association goes to to Professor Claude Lecomte from Nancy Université and CNRS, France.

Claude Lecomte is recognised for his contributions to the measurement and analysis of high resolution X-ray crystallographic charge densities and the insight it has provided. He is acknowledged for pioneering work on high-resolution analyses and charge density modelling of polypeptide and other macromolecular structures based on the analysis of multiple electron density determinations using non-spherical scattering factors. He applied this formalism to hydrogen bonding, porphyrins and metastable states of light-induced spin-transition complexes and to the evaluation of the electrostatic potential from diffraction data on the above and on zeolites, zeolite-like materials and proteins.

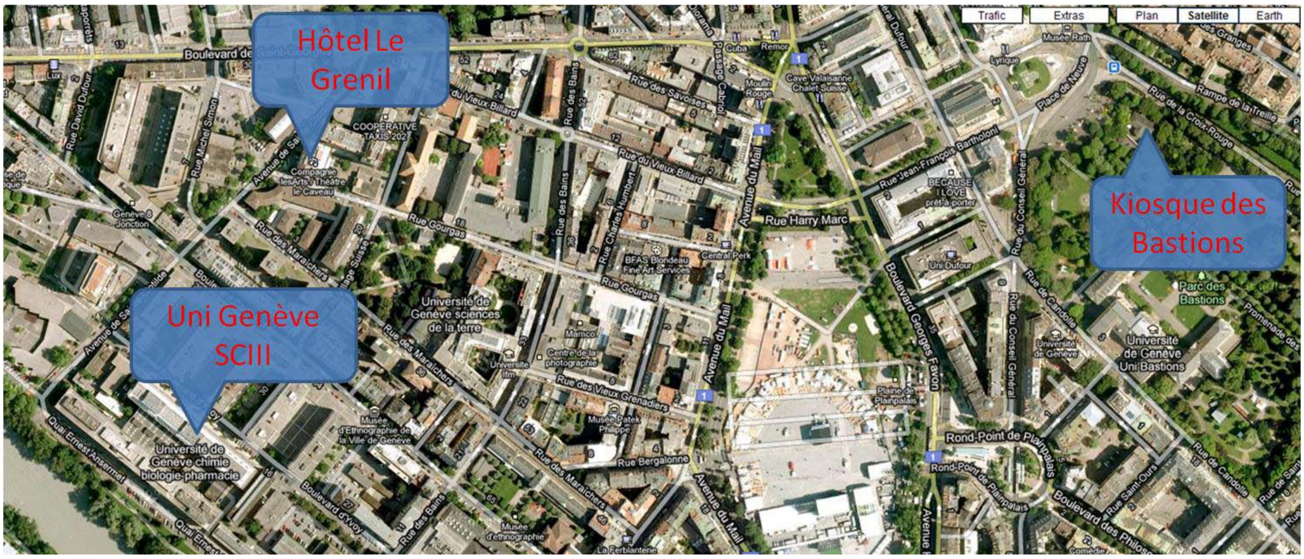
Professor Lecomte will present an acceptance Lecture at the Opening Ceremony of ECM26 in Darmstadt.

SGK/SSCr annual meeting 2010 – Geneva – Sept. 17

The annual meeting of the SGK/SSCR and the general assemblies of SGK/SSCr and SKT 2010 will take place in Geneva on September 17 (meeting and general assembly). It is organized by R. Cerny, Laboratory of Crystallography, University of Geneva.
http://www.unige.ch/sciences/crystal/.

Registration:
In order to optimize our organization, please register free of charge at
http://diffraction.web.psi.ch/sgk-sscr-2010.htm

Dinner: Sept. 16, 2010, **20h** (day before)
Kiosque des Bastions, Parc des Bastions
From the railway station SBB/CFF-Cornavin take the bus no. 5 direction Hôpital, and go out on the 4th station, Place Neuve)
Menu: approx. CHF 55, Registration for the Dinner: Sept. 1, 2010



Meeting Place: September 17, 2010, **08.45h**

SCIII, Lecture Room A 150
Quai Ernest-Ansermet 30, 1205 Genève


Jonction Bus Stop

We are here (Sciences III)

Ecole de Medicine Bus stop

Parking

Sponsors: PANalytical, Bruker, Swiss Academy of Science, SGK/SSCr, University of Geneva, Paul Scherrer Institut

Program of the annual meeting of the
Swiss society for crystallography

New Materials for Clean Future

September 17, 2010, SCIII, Lecture Room A 150
Quai Ernest-Ansermet 30, 1205 Genève

8h45 – 9h15 Registration + Poster hanging
9h15 – 9h20 Welcome (K. Fromm, R. Cerny)

9h25 – 10h10 **Celine Lichtensteiger (University Geneva)**
"Artificially Layered Oxides and Functional Oxide Interfaces″

10h10 – 10h40 Coffee Break / Posters

10h40 – 11h25 **Torben Jensen (University Aarhus)**
"Hydrogen – a Possible Future Energy Carrier ‟

11h25 – 12h10 **Artur Braun (EMPA)**
"Some Applications of Synchrotron and Neutron Scattering and Spectroscopy to Energy Materials Research″

12h10 – 13h00 Lunch

13h00 – 14h00 Annual Meeting of the Swiss Society for Crystallography

14h00 – 14h45 **Michel Latroche (CNRS)**,
″Metal Hydrides as Battery Materials for the Future”

14h45 – 15h30 **Christian Masquelier (University Amiens)**
" Li-ion Batteries : Polyanions at the rescue"

15h30 – 16h15 Coffee Break / Posters

16h15 – 17h00 **Rainer Hock (University Erlangen)**
"Photovoltaic Cells, Cheap Energy for Everybody? "

17h00 – 17h45 **Christian Serre (IL Versailles)**
‟ *Bigger and Bigger Pores, More and More Gas to Store in?* ″

17h45 – 18h00 Poster Prizes and Closing Remarks

Agenda of the SGK/SSCr General Assembly 2010

September 17, 2010, SCIII, Lecture Room A 150, **13-14h**
Quai Ernest-Ansermet 30, 1205 Genève

a) Jahresbericht
le rapport annuel

b) Jahresrechnung
les comptes annuels

c) Aufstellung des Budgets für das kommende Jahr/
le budget proposé pour l'année suivante

d) Festsetzung des jährlichen Mitgliederbeitrages/
le montant de la cotisation annuelle

e) Wahlen/Elections:

1) Ersatzwahl für Prof. Klaus Yvon
2) Wahl eines weiteren Mitglieds für den Vorstand

Vorschläge können dem Vorstand schriftlich mitgeteilt werden respektive direkt an der Generalversammlung angebracht werden.
Eventuelle Kandidaten werden gebeten, sich kurz vorzustellen.

f) Anträge von Mitgliedern
Motions des membres

Quorum for final decisions: ≥ 10%, out of 168 members (148 regular, 20 students)

Abstract Invited Talks

T1: Artificially Layered Oxides and Functional Oxide Interfaces

Céline Lichtensteiger

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Since 1986, remarkable developments in epitaxial oxide growth have taken place, initially motivated by the discovery of high-Tc superconductivity.

It is today possible to grow atomically flat oxide thin films, heterostructures and superlattices with high crystalline and surface quality [1]. Additionally, one can combine oxides with vastly different electronic properties, ranging from superconductors, metals, and ferromagnets to dielectric, ferroelectrics, multiferroics, and Mott insulators in fully epitaxial structures.

These developments yielded to the creation of artificial oxide superlattices and complex heterostructures that are exciting systems where one hopes to develop controlled properties and/or new functionalities.

We will mention a few examples of interesting systems including the conducting and superconducting $LaAlO_3/SrTiO_3$ interface [2] before to focus on $PbTiO_3/SrTiO_3$ superlattices where interactions at the interfaces lead to a new type of ferroelectricity [3].

[1] Ahn et al., Science 303, 488 (2004)
[2] Caviglia et al., Nature 456, 624 (2008); Reyren et al., Science 317, 1196 (2007)
[3] Zubko et al., Phys. Rev. Letters 104, 187601 (2010); Bousquet et al., Nature 452, 732 (2008)

T2: Hydrogen – a Possible Future Energy Carrier

Torben R. Jensen

Center for Materials Crystallography, Center for Energy Materials, Interdiciplinary Nanoscience Center, Department of Chemistry, Aarhus University, Denmark

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The global need for energy is expected to increase by 45 % within 2006 to 2030 [1], which may lead to energy security problems and climatic problems. The present energy supply is based on the limited resource fossil fuels, the use of which is causing increasing CO_2 content in the atmosphere, which may lead to global warming, rising sea levels and more frequent and extreme weather events.

Renewable energy in the form of solar, wind or wave energy is an alternative inexhaustible resource, but its utilisation is hampered by its fluctuation in time and non-uniform geographical distribution. The solution is a safe, cheap and efficient energy carrier, and hydrogen is a world-wide target receiving increasing political and scientific interest. Unfortunately, safe, efficient and economic storage of hydrogen allowing it to be the successor of gasoline is still lacking. This problem has prompted extensive efforts to develop solid state hydrogen storage systems, i.e. a variety of metal hydrides have been investigated as possible hydrogen carriers.

In this talk energy security and environmental problems will be discussed by comparing the present use of fossil fuels with a possible future hydrogen based energy system. A wide range of scientific and technological problems remains to be solved before this can be a reality. It will be illustrated that novel discoveries within nano-materials-science, which can form the basis for new technologies may be our only hope to create a sustainable energy system in future. Within this scenario *Powder X-ray Diffraction* is the most important, versatile and informative technique, which will be underlined by many examples from our recent research.

The goal of our research is to develop new classes of solids for hydrogen storage. We will report on our recent results within three different experimental approaches (*i*) synthesis of novel materials, (*ii*) tailoring materials properties, e.g., by anion substitution and (*iii*) nano-confinement of hydrides and chemical reactions. We have recently synthesized and investigated several series of double metal borohydrides, $M–M'–BH_4$ typically containing an alkali metal, M, and another metal e.g. M' = Mg, Ca, Sr, Ba, Sc, Y, Zn or Cd, and have discovered a fascinating structural chemistry within borohydride chemistry [2-4]. We have also prepared several solid solutions by anion substitution $BH_4^- \rightarrow X^-$, with varying compositions, e.g. $Li(BH_4)_{1-x}X_x$ and $Ca((BH_4)_{1-x}X_x)_2$, $X^- = Cl^-$, Br^- or I^- [5-6]. Nanoconfined chemical reactions have a high degree of reversibility and stability and possibly also improved thermodynamic properties as compared to bulk conditions. This new scheme of nanoconfined chemistry may have a wide range of interesting applications in the future, e.g. within the merging area of chemical storage of renewable energy [7-8].

[1] IEA, world energy outlook 2008.
[2] A Series of Mixed-Metal Borohydrides D. Ravnsbæk, *Angew. Chem. Int. Ed*. 2009, **48**, 6659–6663.
[3] $NaSc(BH_4)_4$: A novel scandium-based borohydride, R. Černý, et al. *J. Phys. Chem. C*, 2010, **114**, 1357–1364.
[4] Thermal polymorphism and decomposition of $Y(BH_4)_3$, D. Ravnsbæk, et al. *Inorg. Chem*., 2010, **49(8)**, 3801-3809.
[5] Anion substitution in $Ca(BH_4)_2$-CaI_2: Synthesis, structure and stability of three new compounds, L. Rude, et al. *Inorg. Chem*., 2010, subm.
[6] Structure and dynamics for $LiBH_4$-LiCl solid solutions, L. Mosegaard, *Chem. Mater*., 2009, **21**, 5772-5782.
[7] A Nanoconfined Reversible Chemical Reaction, T. K. Nielsen, et al. *ACS Nano*, 2010, In press.
[8] Confinement of MgH_2 nanoclusters within nanoporous aerogel scaffold materials. T. K. Nielsen, et al. *ACS nano*, 2009, **3(11)**, 3521–3528.

T3: Some Applications of Synchrotron and Neutron Scattering and Spectroscopy to Energy Materials Research

Artur Braun

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I would like to use the opportunity to show a number of x-ray based and neutron based analytical techniques which can help us to a little better understand the functionality of batteries, fuel cells, capacitor and maybe solar cells, and their correlation with their morphology, and crystallographic and electronic structure. Emphasis is laid on practical examples, and not on theory.

For the structural analysis, I assume the audience is familiar with the well known x-ray (or maybe neutron) diffraction (XRD). Based on that, I want to show some “derivatives” of XRD, and this is resonant XRD in order to obtain some element specific crystallographic information from diffraction. An example is a recent work we did on a ceramic proton conductor material BaZrY-oxide, where we wanted to distinguish a functionality with respect to Zr, and the Y-dopant. Another "derivative" of XRD is small angle scattering with x-rays or neutrons (SAXS, SANS). It is related to light scattering and helps us, with a good statistical robustness, to quantify the microstructure and mesostructure of disordered or amorphous materials. The examples I can show here are porous carbon for a supercapacitor application and for diesel fuel combustion products (soot), and for structural gradients in ceramic fuel cell assemblies before and after operation. Maybe I find the time to bring two more examples, and this is the change of the microstructure in a lithium battery cathode during cycling, and the Ostwald ripening of cobalt nanoparticles in aerogel matrices for Fischer-Tropsch catalysis. A somewhat far-fetched analogy for the "disorder" aspect in scattering techniques is the final example in this talk, and this is quasi elastic neutron scattering (QENS). Here, too, we look at the shape or broadening of signals, but not in order to derive a topological parameter, but in order to learn about the dynamics of a diffusion species in solid matter. QENS allows us to measure the diffusion constant and ionic conductivity at the atomic scale in proton conductors.

In the next part of the presentation we will learn about x-ray and photoelectron spectroscopy and their relevance for the electronic structure of materials. Many physical or chemical properties of materials, i.e. their functionalities for applications, are reflections of their electronic structure. A number of examples include the hard x-ray absorption such as in batteries or fuel cells, where we can monitor the change of oxidation states of 3d metals. This method can tell us a lot about the chemistry and molecular environment of catalyst materials in concert with other gases, such as the sulfur poisoning of Ni by natural gas in a fuel cell anode. We then will move to soft x-rays, particularly to the near edge x-ray absorption fine structure of oxygen in oxides, where we can observe how doping and substitution in iron perovskites can form electron holes in the valence band and thus dramatically change the transport properties of the materials, which is an important aspect for cathode materials. We see immediate correlations of the spectral weight of particular electron transitions and the DC conductivity across doping level and also temperature increase. I hope I find the time to show also how we can in-situ or operando monitor structural and electronic defects with resonant photoemission in some oxide materials which are relevant for solar cell and photoelectrochemical cell applications.

T4: Metal Hydrides as Battery Materials for the Future

Michel Latroche
Institut de Chimie et des Matériaux de Paris Est, CNRS-UMR7182, FR – 94320 Thiais

Metal hydrides are well-known compounds able to react reversibly with hydrogen at ambient pressure and room temperature. They have been extensively studied as potential materials for solid state storage of hydrogen gas. However, those compounds can be formed using two different routes: *via* solid gas or electrochemical reactions. From the second one, electrode materials were developed and so far, the best commercial achievement for metal hydrides has been found in rechargeable Ni-MH batteries that are used worldwide. Nowadays, industry is facing an increasing need for enhanced battery systems able to fulfil very demanding applications such as electric vehicles (EV). For such electrochemical applications, material performances are very challenging. The compounds must exhibit high reaction rate for rapid charging and high power supply. They should sustain thousands of cycles often involving sharp structural phase transitions. Finally, they should be able to resist to highly corrosive alkali medium.

For about two decades, Ni-MH batteries have used $LaNi_5$-based compounds that exhibit excellent properties as negative electrodes. However, they exhibit low capacities (only 1.2 wt.% hydrogen). To improve their weight capacities, such $LaNi_5$-type compounds can be alloyed with $MgNi_2$-type phase leading to so-called superlattice alloys that are now used in commercial batteries. However, real breakthrough will not be achieved without getting rid of heavy rare earths elements. Therefore, the search for new metal hydrides has focused on Mg-rich alloys, since pure Mg has a very high reversible storage capacity (7.6 wt.%). Extremely sluggish sorption kinetics prevents practical application of MgH_2 as a storage medium, but Mg-M (M=Sc,Ti) alloys have recently proven to be viable reversible hydrogen storage media. They were shown to have extremely high reversible electrochemical storage capacities up to more than 5 wt.%, with discharge kinetics far superior to those of pure MgH_2. Finally, the conversion reaction between Mg or Ti hydrides and lithium metal is foreseen as a possible route for new negative electrodes for Li-ion batteries.

For all these compounds, their use as electrode materials implies solid state reactions, phase transitions, defect generation and surface corrosion. To optimize them, one needs a careful investigation of their crystal structure either in the pristine state or during *in situ* operation. Using different probes (X-ray, neutrons), their electrochemical behaviour are studied in close relation with their structural properties. They are now investigated and optimized to develop battery materials for the future

T5: Li-ion Batteries : Polyanions at the Rescue

Christian Masquelier

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Phosphate-based framework structures are serious contenders as the positive electrodes of choice for Li-ion batteries, in particular for the development of safer, cheaper and long lasting energy storage systems desperately needed for electric vehicles.

Aqueous routes used for the synthesis of nanoscale particles of $LiFePO_4$ may lead, under specific conditions, to a full solid solution behavior upon Li^+ extraction at ambient temperature, hence favoring an easier monitoring of the state of charge/discharge of the electrode [1]. We demonstrated through neutron diffraction that this behavior is strongly connected with the presence of significant amounts of structural defects within the crystallites (Li ⇔ Fe exchange, vacancies) [2]. We demonstrated as well that it was possible to monitor and adjust the amount of structural defects through careful annealing in air between 140°C and 500°C, up to a composition close to $LiFe_{0.67}PO_4$ [3]. An important feature is that these findings suggest unusually high mobility of Fe within the triphylite framework.

The resulting powders show complete new electrochemical features at various redox steps between 3.5 V and 2.8 V vs Li, associated with partial redistribution of Fe and Li within the octahedral sites. New crystal super-structures with long range ordering of Fe, vacancies and/or Li, determined through high resolution Synchrotron X-Ray, neutron and electron diffraction [4]

[1] F. Mestre-Aizpurua, S. Hamelet, C. Masquelier & R. Palacin ; *J. Power Sources,* 195(19), 6897-6901 (2010)
[2] P. Gibot, M. Casas-Cabanas, L. Laffont, S. Levasseur, P. Carlach, S. Hamelet, J-M. Tarascon and C. Masquelier, *Nature Materials*, 7, 741-747 (2008)
[3] S. Hamelet, P. Gibot, M. Casas Cabanas, D. Bonnin, C. Grey, J. Cabana, J. B. Leriche, J. Rodriguez Carvajal, M. Courty, S. Levasseur, P. Carlach, M. Van Thournout, J. M. Tarascon & C. Masquelier ; *J. Mater. Chem.,* 19, 3979-3991 (2009)
[4] S. Hamelet, M. Casas Cabanas, L. Dupont, C. Davoisne, J.M. Tarascon & C. Masquelier, submitted, August 2010

T6: Photovoltaic Cells, Cheap Energy for Everybody ?

Rainer Hock

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The efficiency of the primary conversion of photons into electric energy via the photoelectric effect solely depends on the absorber material used. If the theoretically predicted and technologically expected structural and physical properties of the absorber like the band-gap, charge carrier mobilities and finally the conversion efficiency can be reached with a specific solar module, depends as well to a great extent on the quality and homogeneity of the absorber material produced. Since the absorber layer still contributes about 30% - 50% to the final solar module costs, the search for cheap production routes of inexpensive and nontoxic materials with highest efficiencies achievable, is a key issue for cost reduction on the way towards an affordable and environmentally acceptable energy supply for everybody.

At the heart of solar cell fabrication therefore is the knowledge of crystal growth, either of single crystals like for monolithic Silicon based cells, or of polycrystalline thin films of substances with increasing crystallographic and chemical complexity like Chalcopyrites and Kesterites or even more structurally complex sulfosalts.

In this presentation, examples of thin film crystallization techniques for the Chalcopyrites and Kesterites and some of their pros and cons with respect to the fabrication of the different materials will be discussed. For these materials, the crystallization of thin absorber films proceeds via a multitude of parallel and successive solid state reactions, accompanied by melt phases and sometimes peritectic phase transitions. The corresponding ternary to pentanary phase diagrams are rich in phases – if completely known today at all - and crystallization may proceed by different reaction paths leading to different material properties. Time resolved powder diffraction is one versatile tool to follow the crystallization routes taken and to understand and optimize the thin film absorber formation. Examples of powder diffraction measurements during the crystallization of Chalcopyrite and Kesterite thin films will be presented.

For absorber films produced by the reaction of metallic thin films with selenium and sulfur (in the stacked-elemental-layer-process), the crystallization event may be seen as high temperature corrosion of the metal film in a chalcogen atmosphere. A comparison will be made between existing knowledge about corrosion mechanisms and the thin film production for solar cell materials. Models of corrosion can predict and explain some pecularities found in the crystallization of thin film solar cell absorbers.

T7: Bigger and Bigger Rores, More and More Gas to Store in ?

Available at the conference

Abstract Poster

P1: Evolution of the Twin Domain Structure of $SrFeO_{2.5}$ Single Crystals with Temperature Observed by Fast Data Acquisition on ID11 @ ESRF

Ravi Sura[1,2], Monica Ceretti[1], Olivier Hernandez[1], Carmelo Prestipino[1], Juerg Schefer[2], Lukas Keller[2], Gavin Vaughan[3] Werner Paulus[1]

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[2] *Laboratory for Neutron Scattering (LNS), Paul Scherrer Institut,*
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Oxides with high oxygen ionic conductivity have attracted considerable attention in the last decade. This is mainly related to their potential application as electrodes for solid oxide fuel cells or batteries and membranes for oxygen separation. The two structure types know today to undergo oxygen intercalation at ambient temperature belong either to the K_2NiF_4 structure, e.g. $La_2MO_{4+\delta}$ (M = Co, Ni, Cu) or to the deficient perovskites and more specifically to the Brownmillerite-type structure such as $SrMO_{2.5}$ (M = Fe, Co).

To better understand the underlying mechanisms for oxygen mobility in the Brownmillerite-type frameworks, high quality single crystals have been obtained by the floating zone method. We subsequently investigated the structural evolution of orthorhombic $SrFeO_{2.5}$ with the uptake of oxygen as a function of temperature. Fast and high quality data acquisition has been obtained on the ID11 beamline at ESRF, equipped with a 2D Frelon camera. We focus here especially on the complex evolution of the twin domain structure of the $SrFeO_{2.5}$ crystal with the uptake of oxygen at different temperatures in air.

P2: Using Short Wavelength Radiation for Chemical Crystallography

E. Hovestreydt[1], H. Ott[1] and J. Graf[2]

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Combining synthetic multilayer mirrors with micro-focus X-ray sources (rotating or stationary target) has become a standard with in-house X-ray sources for single crystal diffraction as well as a number of applications in powder diffraction[1]. The maximum angle of incidence at which a multilayer mirror reflects is significantly smaller for higher energy radiation, such as Mo-K_α or Ag-K_α radiation than it is for Cu-K_α radiation. This is why synthetic multilayer mirrors traditionally have been used for Cu-K_α radiation or softer wavelengths. Modern deposition technology, however, allows for the reproducible production of high quality multilayer mirrors with smaller *d*-spacing. In consequence these mirrors reflect higher energy radiation at larger angles of incidence [2, 3]. Combined with the latest generation of reliable and stable micro-focus tubes this provides new high-performance low-power X-ray sources for shorter wavelengths.

microfocus performance for solid state chemistry

We will present selected results on the use of these high-performance sources. In particular we will address the use in small molecule and high-pressure crystallography.

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P3: HRXRD Studies on High Quality Epitaxial Germanium on Pre-patterned Silicon

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An innovative germanium-on-silicon growth technology has been established. Thick germanium layers can be grown with an exceptionally high quality on patterned silicon. The micro-machined Si single crystal pillars serve as substrate for epitaxial growth of thick Ge layers having thicknesses up to 20μm (Fig. 1, left).

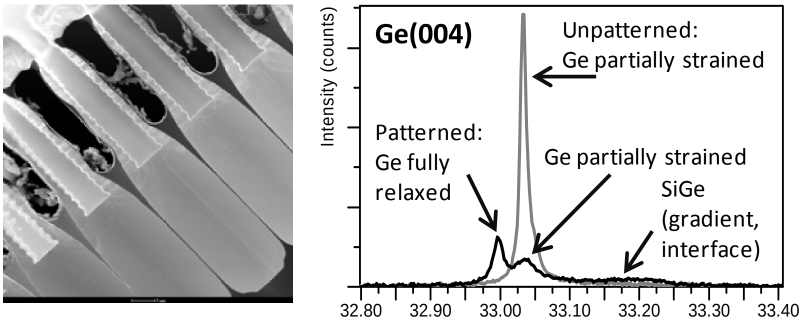

Fig. 1 *STEM view of 8 μm Ge towers on Si pillars (left) and high resolution omega/2theta scans (right) on a 8μm thick Ge layer on a patterned Si substrate (black) and on a unpatterned Si substrate (grey).*

The Ge layers were grown epitaxially on Si(001) by low-energy plasma-enhanced chemical vapor deposition (LEPECVD) using exceptionally high growth rates and low substrate temperatures of the order of 500^o C.

The Ge/Si system shows a lattice mismatch of about 4.2 % and in addition, a high mismatch of thermal expansion coefficients. The deposition of germanium layers results in a high strain and therefore often in wafer bowing and Ge layer cracking. The high density of misfit and threading dislocations can significantly degrade the performance of a device.

HRXRD methods are used to evaluate lattice strain and relaxation, lattice tilt, layer composition and dislocation densities. Single scan simulations on symmetric (004) and asymmetric (115) reflections are performed in order to obtain precise information about the Ge/Si system. The degree of relaxation can be determined with highest precision using the reflections from the Si substrate as internal reference. In addition, Reciprocal Space Maps (RSM's) are carried out to give a detailed view on lattice strain and tilt but also defect densities.

The figure 1 (right) shows the high resolution ω/θ scans carried out on the Ge(004) reflection of a sample having a patterned Si substrate and for comparison of a sample having an unpatterned Si substrate. While the latter is strongly strained, complete relaxation is observed for the germanium epitaxially grown on the Si pillar substrate. The threading dislocation density is reduced to zero and a perfect crystal quality is obtained despite the large lattice mismatch. No layer cracking and a minimized wafer bowing is observed which presents an innovative key technology to wafer handling and post-growth processing.

P4: Silver Complexes and Nanoparticle Formation with Polymers

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Nowadays, a wide variety of composite materials consisting of polymers containing metal nanoparticles have been extensively investigated to realize their potential applications ranging from optoelectronics to biomaterials. Nanostructured materials consisting of silver nanoparticles (Ag-NPs) embedded in polymeric matrices show physico-chemical, optical and antibacterial properties [1, 2]. A simple method to prepare a AgNP/polyelectrolyte composite was successfully used. Thus, Ag-NPs were obtained through spontaneous formation of nanostructured silver from an Ag_2O/polystyrene sulfonate (PSS) solution. The kinetics of AgNP-formation was investigated by dynamic light scattering and UV/vis spectroscopy, and related morphology was investigated by X-ray diffraction techniques. The synthesis of the Ag-NP/polystyrene sulfonate composite was performed by mixing a PSS solution in water with a Ag_2O solution in diluted ammonia during 48 hours. The nanoparticle size was determined by TEM and SEM. Silver complexes with different types of ligand show also interesting antibacterial properties. Tünde Vig Slenters[3] investigated the properties of ethylene glycol derivatives with silver. Now we investigate polyethylene glycol complexes.

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P5: Precursors for Oxide Materials

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Heterometallic compounds can be used in different applications, for instance as precursors for opto-electronic devices or mixed metal oxides in high-T_C superconductors (e.g YBCO) [1, 2]. A synthetic strategy to obtain mixed metal oxides is to use decomposition techniques like (metal-organic) chemical vapour deposition (MO-CVD) to combust metal-containing volatile complexes. Complexes of alkaline earth, group 11 or also group 1 metal ions may for example provide such precursors. For the drastic decomposition process, it is suggested to use simple ligand systems which can easily be prepared in large scale. Complexes should then easily be obtained.

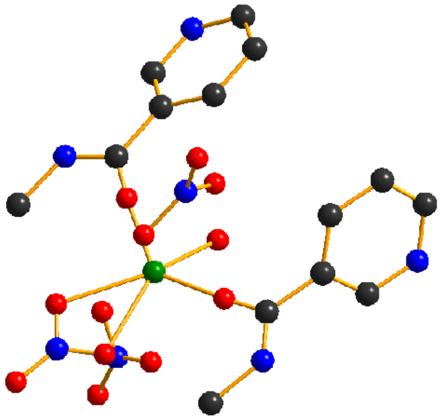

Fig. 1

In this work homometallic complexes with yttrium and copper, but also mixed metal complexes containing both, yttrium and copper, were investigated using cheap and easily preparable ligands. The general reaction scheme is as follows:

$$\text{Ligand} + \text{Metal salt} \xrightarrow[\substack{\text{stirring} \\ \text{RT 24 h}}]{\text{Solvent}} \text{Complex}$$

In the beginning, different ligands (mainly based on nicotinic acid and its derivatives) have been synthesised. All ligands were reacted first with either yttrium or copper ions, then with both. Complexation was often proven by IR, and many efforts were undertaken to obtain single crystals of these complexes. Analyses, like IR, TG or elemental analysis of the other compounds were performed and in the last step some of the complexes were tested on their potential for single-source precursor. Different coating techniques were used, like MOCVD or drop-coating and the obtained thin films and powders were analysed by PXRD and SEM/EDS.

Among all reactions, it was possible to identify a new yttrium precursor $[Y[C_7H_8N_2O]_2[NO_3]_3H_2O]$ (Fig. 1). This compound was found to decompose already at 150°C to yttrium oxide, which is a very elegant way to access Y_2O_3 compared to literature where 460°C during 12 hours are required to decompose yttrium oxyhydroxide [3].

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P6: Synthesis of New Silver Compounds with Nicotinic acid DerivativesTransformations

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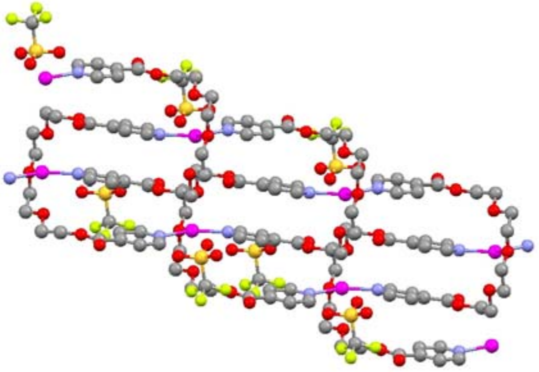
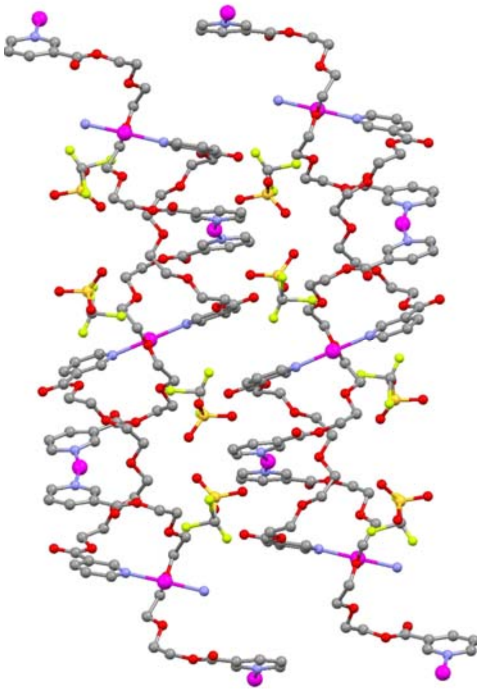
Complexes with silver ions have a great potential for applications in medical uses. Those complexes with ligands derived from nicotinic acid and with polyethylene oxide groups as spacer are synthesized.
Upon coordination to silver ions, polymorphism can be observed: metallacycles, helix and, under special conditions, chain structures are obtained.





Finally we propose the development of complexes with longer spacers to study the possible formation of liquid crystals

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CrytEngComm, **8**, 403-416, (2006)

P7: Penta-coordinate Cr(II) as water reductant and starting compound for new Cr(III)-clusters and coordination polymers

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The use of alkali aryloxide reagents in organo-metallic synthesis often depends on their solubility, a property derived from their structure. The regain of interest of alkali aryloxides also originates from the discovery of high-temperature superconducting compounds, which has generated a great interest in the formation of oxide materials and other ceramics. Thus, many alkoxides of yttrium and copper are common precursors for oxide materials. Moreover, the synthesis of heterobimetallic alkoxides has provided a facile route for obtaining soluble, volatile, and generally monomeric species. These heterobimetallic complexes can thus serve as valuable precursors for making metal oxides but it is not the only possible application for this type of compounds. These complexes can be used as starting compounds for syntheses of more complex structures.

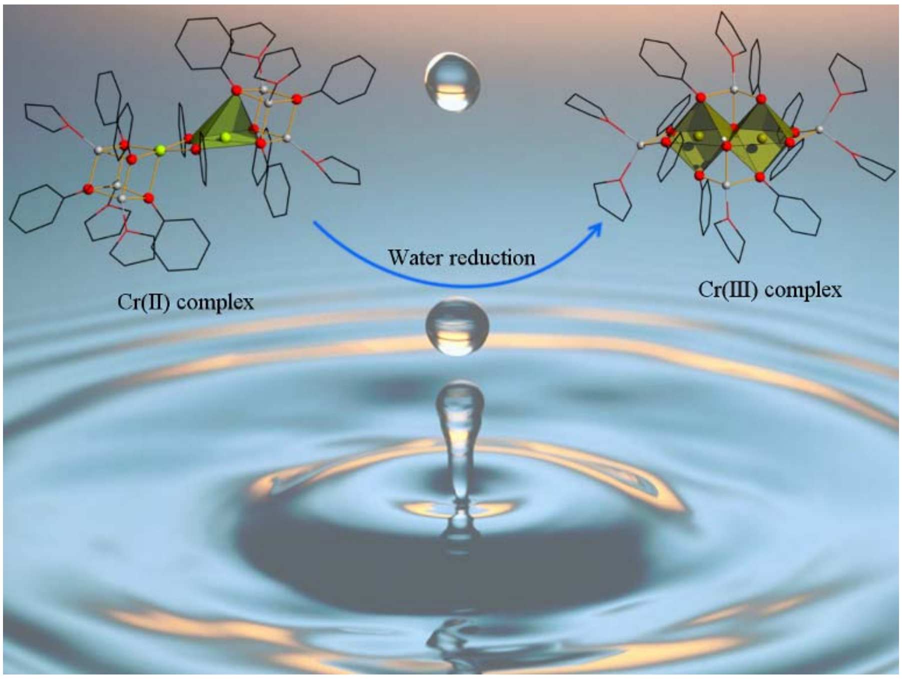

Figure 1:
Oxidation of a Cr(II) complex by water and formation of a new Cr(III) complex family.

In this context, with a penta-coordinate Cr(II) complex synthesis by *Edema* [1], we have synthesized and characterized some new Cr(III) aryloxide complexes with the highlight being the oxidation of the chromium(II) to chromium (III) [2].

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P8: Nano-structured Manganese Based Olivine Cathode for High Power Lithium Ion Batteries

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Lithium ion batteries have played a key role in the portable electronic revolution and the technology is now being vigorously pursued for vehicle applications.

The use of nanosized particles of cathode materials is needed in order to obtain rapid charge and discharge reactions for high power lithium ion batteries applications. To enhance these charge and discharge reactions, Li-ion diffusion path in the nano-structured cathode should be short. [1, 2]

Nano-sized $LiMnPO_4$ material is prepared via two types of wet chemical methods. Water-based direct precipitation method provides well-crystallized $LiMnPO_4$ with various shapes depending on volumetric ratio between water and co-solvent. The other method using surfactant provides much smaller particle size (30 ~50 nm) with spherical shape

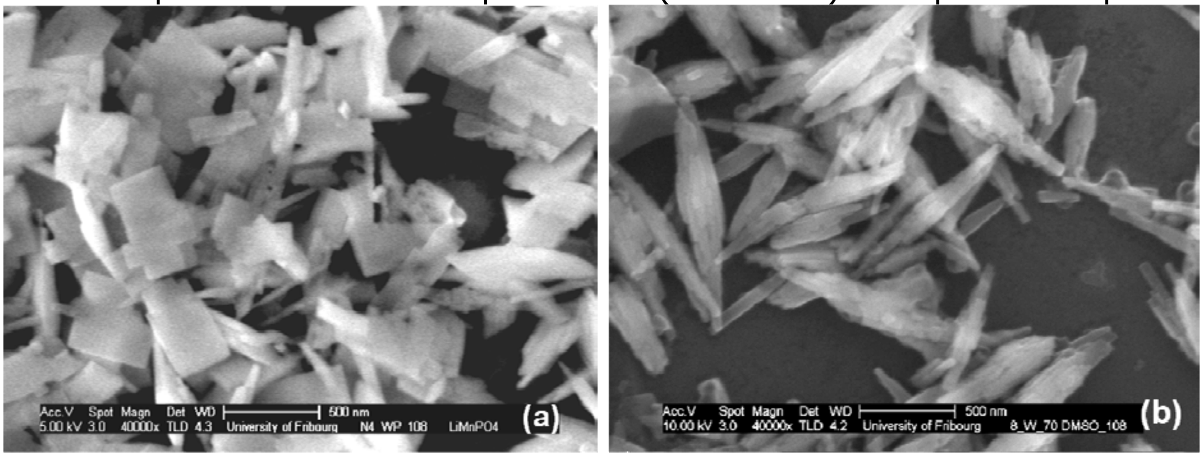

Fig. 1. Scanning electron microscopy images of $LiMnPO_4$ particles prepared by water based direct precipitation with water : DMSO ratio to (a) 50:50 vol% and (b) 30:70 vol%.

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P9: Oppechowski-Guccione-like Symbols of Magnetic Space Groups

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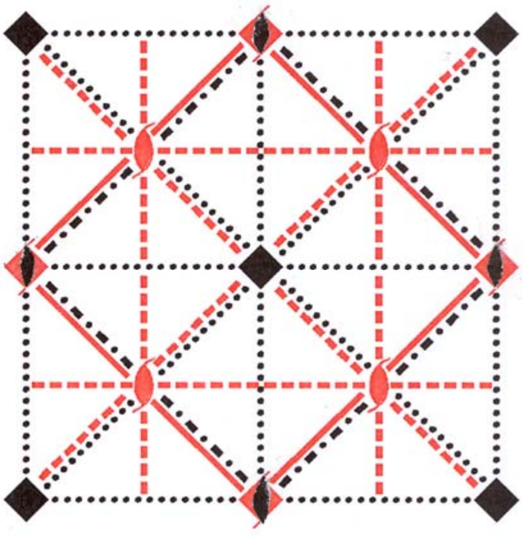
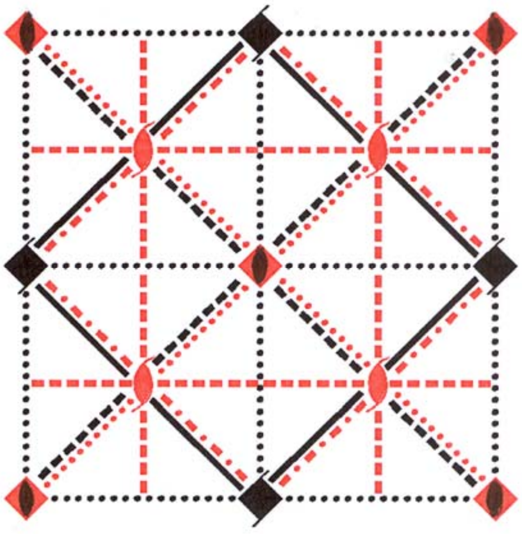
For the magnetic space group types with black and white lattice two sets of symbols have been proposed: the BNS symbols [1] and the OG symbols [2]. Whereas generators of the group can be read off the BNS symbol, the International Tables for X-Ray Crystallography (1952) must be consulted to interpret the OG symbols in the cases where the black and white lattice is centred. We define OG-like symbols in the case of centred lattices in such a way that generators of the group can be deduced directly from the symbol [3]. The definition generalizes a proposal of Bertaut [4] for crystal class *mmm* to every crystal class.



Symmetry diagram for the space group with
BNS symbol P_I4cc
OG-like symbol proposed in [3] I_P4cc
OG symbol according to [2] $I_P4c'm'$



Symmetry diagram for the space group with
BNS symbol P_I4_2cm
OG-like symbol proposed in [3] $I_P4'cc'$
OG symbol according to [2] $I_P4'c'm$

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P10: Ionic Conductivities in Solid State Crown Ether Channel Systems

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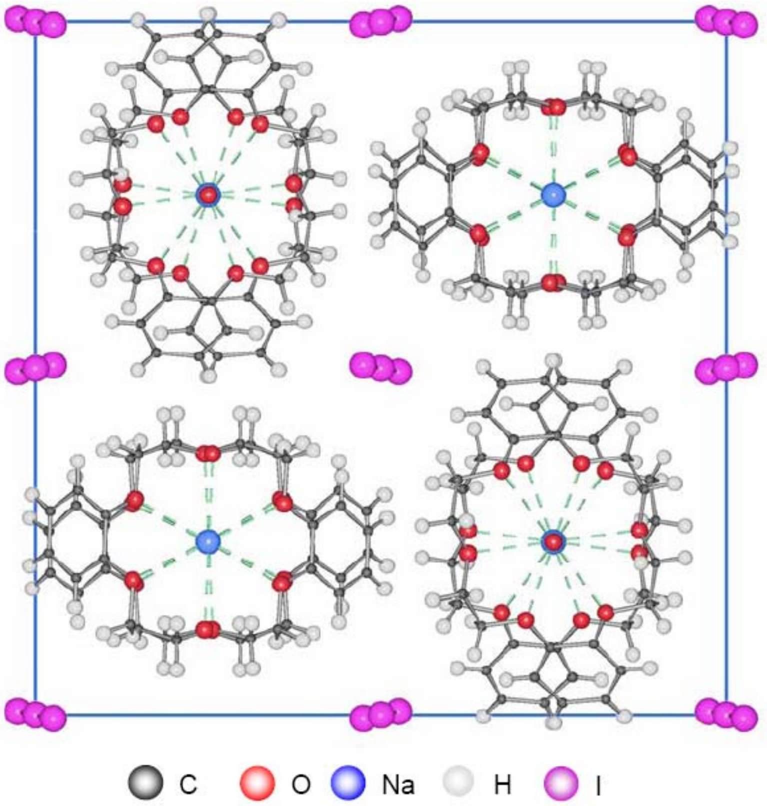

Figure1. Unit cell of sodium compound , view down the c-axis of the channels

The potential miscellaneous applications of artificial compounds mimicking the conductivity properties of ion channel proteins have stirred considerable efforts towards developing such compounds. Channels are ubiquitous in living systems and channels are often "gated" to open only under specific conditions [1]. The design of artificial channels draws on the accumulated expertise in the field of supramolecular chemistry while their study offers the potential for revealing new facets of the physical chemistry of ion transport. Monomeric species capable of traversing a membrane may self-associate to form an oligomeric pore in the manner of amphotericin [2, 3]. Several polymers may be pre-organised by attachment to a central core. The latter are crown ethers and calix[n]arenes. They are the bases for the approach to artificial ion channels (Figure1) [4, 5].
The aim of this research is to use dibenzo-18-crown-6 as scaffold to build artificial aquaporins and potassium ion channels and else, to study sodium ion and proton transport through these polymers.

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P11: Local Atomic Order in Apparently Disordered Crystals: What we Can Learn from the Pair Distribution Function

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Many metallic alloys, intermetallic compounds and metal hydrides show disordered crystal structure (partly or mixed occupied and/or split or close atomic sites) when Bragg scattering is analyzed by the Rietveld method. If the crystal appears from Bragg scattering as disordered, it does not mean that it cannot be ordered locally. Significant, and often sufficient, information about the local order can be gleaned from the analysis of the atomic Pair Distribution Function (PDF). We will show on two examples what is possible to learn from the PDF obtained from a powder sample:

The crystal structure of $YCu_{6.576}$, a substitution variant of the $CaCu_5$- type, was shown to contain Cu_2-dumbbells replacing Y-atoms in a fraction s=0.19 in the formula $Y_{1-s}Cu_{5+2s}$. The structure type $TbCu_7$ is assigned to the disordered structure. The local order in the vicinity of the Cu_2-dumbbells has been derived from the PDF obtained from the X-ray powder data [1]. The coordinating Cu_6-hexagon around the dumbbell site shows a shrinkage of 0.33(1) Å *w.r.t.* the equivalent environment of the Y-atom. No adjacent Y-atoms are substituted but a hexagonal arrangement of Cu_2-dumbbells exists in layers perpendicular to **c**. The stacking along [001] is random and can be modeled locally from the PDF data consistent with both *ab*- and *abc*-type stacking. Therefore, the local order is comparable to that in $Ni_{17}Th_2$- and Th_2Zn_{17}-structure types, ordered variants of $TbCu_7$. These results agree with the atomistic simulations based on interatomic pair potentials extracted from *ab-initio* calculated total energy curves by adopting modified Möbius inversion method [2].

Deuterium local order in cubic Laves phase deuteride $YFe_2D_{4.2}$ was studied by neutron (*ToF*) powder diffraction experiments and PDF modeling [3]. The minimal D-D distance in a metal hydride of 2.1 Å (Switendick rule) has been experimentally proved in the HT-disordered phase. The distribution of deuterium atoms around the iron is not random, and cannot be explained only by applying the Switendick rule. The local order of deuterium atoms within the first iron coordination sphere in the HT-disordered phase above 343 K is similar to the iron coordination in the LT-ordered phase. Same composition of the coordination polyhedra FeD_5 and FeD_4 was found in both phases by Reversed Monte Carlo modeling of the PDF. The exact shape of the FeD_5 and FeD_4 coordination polyhedra in the HT-disordered phase cannot be, however, unambiguously revealed by the PDF analysis.

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P12: Precursors for Ion Conducting Battery Oxide Materials

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Ion conducting materials are increasing interest predominantly due to the commercial interest in solid oxide fuel cells and Li batteries. The layered oxide phases of $LiMO_2$ (M = Co, Ni) are often used as cathode materials of these batteries [1, 2]. While the search for new materials continues to attract considerable attention there has been a noticeable increase in research of new precursors for those materials. Metal alkoxides have been proposed like good route precursors due to their high solubility, low decomposition temperatures, cross linking ability, ease of modification and commercial availability [1, 4].

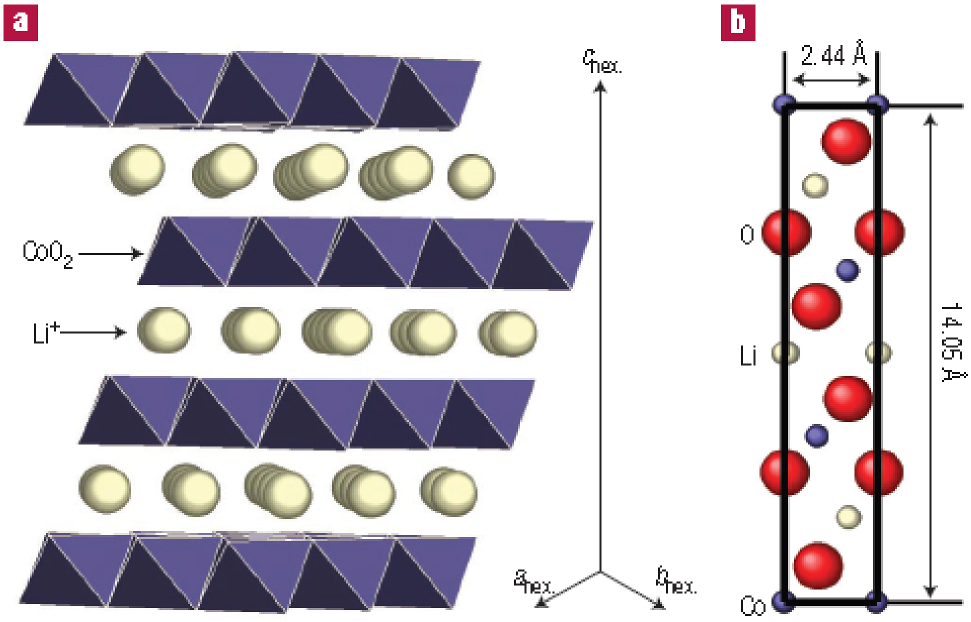

Figure 1: Models of the layered $LiCoO_2$ structure with space group R$\bar{3}$m.
a, Drawn with Li ion horizontally between CoO_2 octahedra.
b, Projection along the [110] zone axis[3].

For this purpose we developed new metal alkoxides materials using chloride of iron, nickel cobalt, and manganese as starting materials. We will present the first result of this work using the Schlenk techniques under Argon flow to synthesize those precursors.

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P13: New Developments in Structure Analysis of Powders using Preferred Orientation

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In recent years, the reflection overlap problem, which is the primary hindrance to structure determination from powder diffraction data, has been addressed by exploiting preferred orientation. The changes in the diffraction pattern intensities as a function of sample orientation (tilt χ and rotation ϕ) that are observed for a textured sample in an X-ray beam, can be used to resolve the relative intensities of reflections that overlap in a conventional powder diffraction pattern. It has been shown that this principle can indeed be exploited to obtain more single-crystal-like data from a polycrystalline material [1]. The availability of a new version of the 1-dimensional Si-microstrip detector Mythen II on the Materials Science Beamline at SLS [2] and new features in the data analysis software *Maud* [3] have made it possible to improve the quality of extracted reflection intensities significantly. Textured samples of a zirconium phosphate framework material (ZrPOF-pyr) with a known crystal structure [4] and of an ammonium niobium silicate with an unknown structure were measured recently using the new experimental setup. In this process 302 orientations are measured. Then data analysis using *Maud* involves two steps. First, the intensities of a few non-overlapping reflections in all 302 patterns as a function of sample orientation are used to generate pole figures (PFs) for these reflections and to determine the orientation distribution function (ODF). The ODF is then used to calculate the pole figure values for all reflections in the pattern, and these are applied in a joint refinement of a selection of the patterns to obtain a single set of single-crystal-like reflection intensities. Analysis of the ZrPOF-pyr data shows that indeed more reliable intensities can be derived for reflections that overlap in a conventional powder diffraction pattern. However, the few orientations corresponding to main zone axes (e.g. *h00, 0k0, 00l, hh0, h0h, 0kk*), which were used first for intensity extraction, do not contain sufficient information to resolve all overlapping reflections. Consequently, a new strategy for selecting an optimized set of sample orientations for intensity extraction has been developed. All PFs are calculated from the ODF, and from these, difference PFs are calculated for overlapping reflections. Those orientations that represent maxima in the PFs (i.e. above a given threshold value) and maximum differences for overlapping reflections (texture contrast) are selected. First results of this new approach using data from our last experimental sessions in May and June 2010 will be presented.

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P14: Complexation Properties of Completely Reduced *meso*-Octamethylporphyrinogens (Calix[4]pyrroles)

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Calixpyrroles bind anions efficiently and can be transformed into transition-metal complexes only under forcing conditions. Reducing the macrocycle creates a ligand that easily forms classical Werner complexes with copper, nickel, palladium, and iron ions. The metal complexes present an array of four directed hydrogen bonds, which specifically bind the counterions [1,2].

The reaction of the reduced macrocycle with iron triflate has produced some surprises. One complex exhibits a phase change at 173 K, transforming from a monoclinic system (Z′ = 1) to a triclinic system (Z′ = 2). The triclinic phase appears to be a twinned crystal, which on heating reverts cleanly to the monoclinic non-twinned crystal. The question is posed, at 173 K is the crystal really twinned or simply split?

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P15: Structure/Property Relationship by High Pressure Studies: The Changes of Linear and non Linear Optical Properties in 4-Amminobenzophenone

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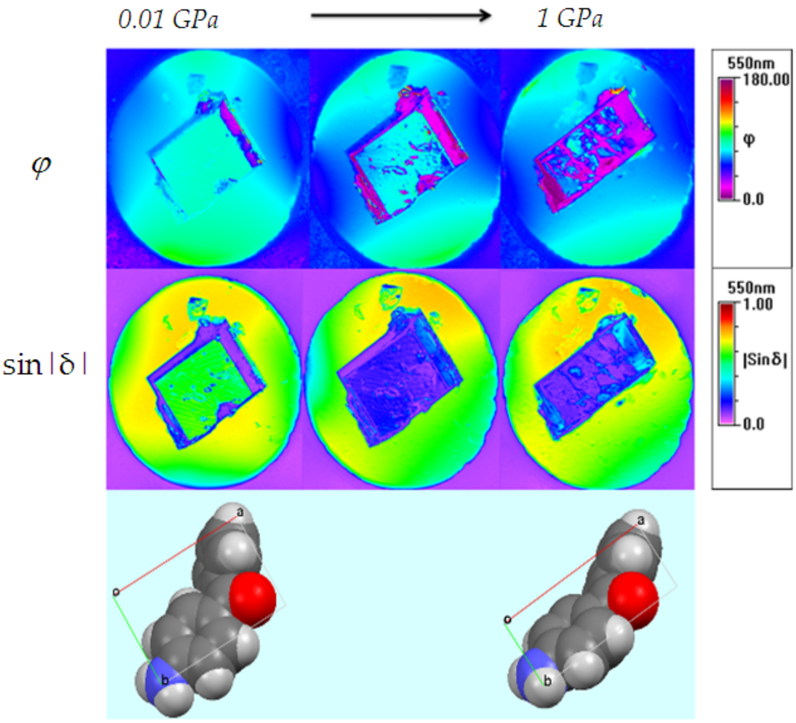
Aim of this work is correlating the molecular and supramolecular structure of second harmonic generation (SHG) crystalline materials to their optical properties, by increasing the pressure. For this purpose we selected the monoclinic acentric ($P2_1$) 4-aminobenzophenone (4-ABP), known to undergo large variations of the SHG activity on increasing the pressure [1], [2].



We have determined several X-ray structures at various pressures to inspect the molecular and supramolecular changes.
At ca. 1 GPa, 4-ABP crystals show a highly anisotropic compression: a and c axis expand whereas b undergoes a large contraction. This is brought into prominence by the polarized light microscopy, using the rotating polarizer method (see the micrographs in the figure). At high pressure, we observed an increased planarity of the 4-ABP molecule, which is the main cause of the anisotropic compression. The supramolecular motif, instead, is characterized by a stacking of the phenyl rings (absent at room conditions).
The reduced torsion between the aromatic rings in a chromophore is known to enhance molecular hyper-polarizability (thanks to the increased conjugation), which in turn is responsible of the SHG signal.
The intermolecular stacking might also be responsible of the more efficient SHG response, as demonstrated by some theoretical work.

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P16: First Biochemical Characterization of the Thymidine Kinase from Leishmania donovani

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Leishmaniasis is a vector-borne parasite disease transmitted to human by protozoan of the genus Leishmania via the bites of the sandflies. The spread of the disease increases concomitantly with the changes in climate and the migration of the insect vector in other new areas. The first line drugs employed to cure leishmaniasis are associated with side effects and toxicity. This considerably limits their use. Moreover, parasites have developed resistance towards the drugs. Therefore, the identification and validation of new targets are crucial to develop new effective treatments. A recent study on Leishmania major and Leishmania donovani, including a genetic validation indicated that Leishmania thymidine kinase could play a role in the virulence of the parasite [1-2], thus could be seen as putative target for a therapeutic intervention using inhibitors.

The Leishmania donovani thymidine kinase (LdTK1) belongs to the type II thymidine kinase family, a family that includes the human cytosolic TK1 (hTK1) [3-4]. Interestingly, LdTK1 possesses a zinc-binding domain within the thymidine kinase domain and harbors at the C-terminus extremity two SH3 domains. LdTK1 shares 51% identity of sequence with hTK1, this guarantees that the fold is conserved but that there are enough differences in the binding site for achieving selectivity with a compound.

The aim of the work is to assess the Leishmania donovani thymidine kinase, LdTK1, as a therapeutic target against leishmaniasis. The ldtk1 gene was cloned and the LdTK1 overproduced and purified in its active form. The first results of its biochemical characterization will be presented.

[1] Thiel et al. Molecular & Biochemical Parasitology 2008;**158**, 152–162
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P17: Effect of Lattice Volume and Strain on the Conductivity of Ceramic Proton Conductors

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[4] Saarland University, Saarbrücken, Germany
[5] School of Chemistry, University of St. Andrews, Scotland, United Kingdom

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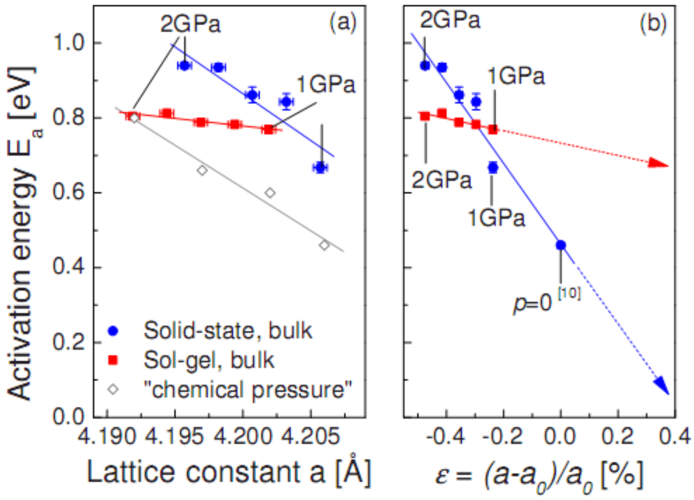

Proton conductors are promising solid electrolyte materials for ceramic fuel cells operating at intermediate temperature. Our study focuses on the molecular mechanisms of proton conductivity of perovskite yttrium-doped barium zirconates and cerates. Our recent results in X-ray and neutron diffractograms of $BaZr_{0.9}Y_{0.1}O_{3-\delta}$ (BZY10) reveal that the onset temperature of lateral proton diffusion coincides with its thermal lattice expansion, which exhibits a contraction for protonated BZY10 at T = 648 K, suggesting a correlation of toughening of the lattice and proton conductivity [1,2]. On the other hand, we found when applying hydrostatic pressures on BZY10 and $BaCe_{0.8}Y_{0.2}O_{3-\delta}$ that the activation energy of proton conductivity decreases linearly with the lattice parameter **(a)** [3-5]. Compressing the lattice decreases proton conductivity, indicating that the protons need "space" to move freely in the lattice. In return, an expanded lattice with strain parameter $\varepsilon > 1$ should have lower E_a **(b)**, suggesting that thin films expansive tensile strain could have larger proton conductivity with desirable properties for applications.

[1] A. Braun, A. Ovalle, V. Pomjakushin, A. Cervellino, S. Erat, W. C. Stolte, and T. Graule, Appl. Phys. Lett. **95**, 224103 (2009).

[2] A. Braun, S. Duval, P. Ried, J. Embs, F. Juranyi, T. Strässle, U. Stimming, R. Hempelmann, P. Holtappels, and T. Graule, J. Appl. Electrochem. **39**, 471 (2009).

[3] S. B. C. Duval, P. Holtappels, U. Stimming, and T. Graule, Solid State Ionics **179**, 1112 (2008).

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P18: The Crystal Structure of D-Ribose Solved from Powder Diffraction Data

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The molecular structure of D-ribose is usually presented as β-furanose, as that is the form it adopts in biochemically important derivatives. However, NMR experiments showed that in aqueous solutions it exists as a mixture of α- and β-furanoses and α- and β-pyranoses, but its crystal structure was not known until recently [1]. In 1956 Furberg et al. investigated crystalline D-ribose [2], but the crystals were of poor quality, so only the unit cell and space group could be derived ($P2_1$, a = 6.509 Å, b = 21.644 Å, c = 4.821 Å, β = 111.0°, Z=4). In view of the advances in structure determination methodology for polycrystalline materials and the importance of ribose in biochemical systems, an attempt to solve its structure from powder diffraction data was undertaken. Data were collected on the MS beamline at the SLS [3] using the Mythen II Si-microstrip detector [4]. Indexing confirmed the unit cell reported fifty years ago. Initially, the powder charge-flipping algorithm [5,6] in *Superflip* [7] was tried, but no solution was found. Then the direct-space global optimization program *FOX* [8] was applied to the problem, even though it was not clear which form the molecule will adopt in the crystalline state. Two molecules in the asymmetric unit, each with 5 possible configurations, give 15 starting models for the optimization and up to 18 degrees of freedom. Several of these were tried using the parallel tempering algorithm with relaxed restraints in *FOX*, but the *R* values of the resulting structures did not differ and a correct solution could not be identified. However, the flexible chain models yielded a distorted U-shape geometry what suggested that the ribose is probably a ring. A long process of generating difference Fourier maps from various starting models was then undertaken. This led to a structure with one α- and one β-pyranose molecule in the asymmetric unit. Refinement converged with the *R* values R_F = 0.081 and R_{wp} = 0.097. Further refinement allowing an α/β disorder in both molecules showed that one molecule was pure β and the other 73% α and 27% β, and resulted in a slight improvement in the *R* values (R_F = 0.077 and R_{wp} = 0.094). This model is consistent with solid state NMR results. As the structure solution was not straightforward, D-ribose is being used as a test case for combining *FOX* and *Superflip*. Preliminary results of this systematic study are promising.

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[8] V. Favre-Nicolin, R. Cerny, *Z. Kristallogr.*, 2004, **219**, 84

P19: Symmetry in Art and Cultural Heritage

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The "Commission on Crystallography in Art and Cultural Heritage" (CrysAC) of the "International Union of Crystallography" (IUCr) was founded in 2008 [1].
The probably most important common topics of art, design, architecture with crystallography, chemistry or materials design are the development and application of new materials, as well as applications of symmetry for the design of objects.
This is reflected in the commission's two main topics:

a) Crystallography and symmetry in art;
b) Crystallographic analysis of artworks and ancient materials.

The first part of the presentation will be a brief introduction of the IUCr "Commission on Crystallography in Art and Cultural Heritage" (CrysAC). The main focus then will be on the symmetry analysis of patterns, applied in the creation of cultural objects, with examples originating from different periods, cultures, and continents.

Up to now only a few systematic approaches in analysing patterns of cultural objects by help of symmetry analysis are on hand; and rarely have they focused beyond the context of mathematics and crystallography [2-3]. The possible power of symmetry analysis for research in archaeology, history or ethnology, as well as its limits still stays mostly unexplored. Through a purely visual approach, symmetry analysis of cultural objects is a truly non-destructive method and could be part of a standard style analysis. It might provide valuable additional information to an art object, its history and cultural context, as well as it could contribute to the history and cultural dimension of scientific knowledge or the history of art-science relationships.

But where would symmetry analysis find its most valuable place? When does the knowledge of a pattern's symmetry provide additional valuable information for archaeologists, ethnologists and art historians? Could we reconstruct historic pattern generation methods, valuable for restaurateurs as well as for contemporary artists and designers? This presentation aims not to answer, but to raise questions, and to foster discussions on possible future applications.

[1] http://www.crystallography.fr/crysac
[2] "Symmetry of Culture", D. K. Washburn and D.W. Crowe, 1988, the University of Washington Press, Seattle
[3] "Symmetry Comes of Age – The Role of Pattern in Culture", Editors D.K. Washburn and D.W. Crowe, 2004, the University of Washington Press, Seattle

P20: Light Hydrides Studied by the Combination of X-ray Powder Diffraction and Theory

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Research in materials science often deals with collecting and interpreting a large amount of powder diffraction data. The data are commonly measured *in-situ*, e.g. in special reaction cells, diamond anvil cells in high-pressure experiments etc, and thus are of a limited quality: such data have a limited resolution both in the reciprocal (relatively broad peaks) and in the direct (limited 2θ range) space. However, these experiments frequently reveal new phases, posing a problem of solving crystal structures from poor diffraction data.
The major help, when using poor data, comes from a parameterization of a structure by building blocks, by introducing less refined parameters, and from a use of the antibump restraints, by limiting the parameter search space. These tools are the empirical ways to take into account energy considerations – the known blocks and the minimum distances represent energy minima in many other known substances. Thus the knowledge of (crystal) chemistry serves as the extra data, complementing the experimental ones.
Experimentalists also seek help from theory. Although the theoretical-only methods mostly fail to predict the most stable structures, the post-experimental DFT-calculations are very useful, helping to correct some details in the experimental models, define positions of hydrogen atoms, orientation of light groups etc. Molecular entities can be first optimized in a gas phase by quantum-chemical methods and defined in a form of Z-matrix, leaving only the torsion angles to be determined in the unknown structure. We see the simultaneous use of total energy estimation together with the global optimization in the direct space with respect to the diffraction data as the most promising development of *ab initio* structure solution. This combination involves two very different cost functions (the fit to the experimental data and the energy minimization), without making presumptions on the molecular geometries, and thus should enable structure solution even from very poor diffraction data.
Recently discovered phases of light hydrides characterized by the combination of techniques include high-pressure phases of $LiBH_4$ [1], ammonia-borane [2], and the novel Li-Al-borohydride complex [3]. The latter compound has a complex composition $Al_3Li_4(BH_4)_{13}$, which can be rationalized on the basis of complex anions $[Al(BH_4)_4]^-$ and cations $[Li_4(BH_4)]^{3+}$, shown on the cover picture. Li-Al-borohydride is a potential on-board hydrogen storage material since it shows decomposition at about 70°C combined with a high gravimetric (17.2 wt%) and volumetric (117 kg/m^3) hydrogen density.

Two microsymposia at EPDIC12 (2010) in Darmstadt, namely MS6 "Structure solution" and MS17 "Materials for energy storage and conversion" are covering the methods and materials mentioned here.

[1] Y. Filinchuk, D. Chernyshov, A. Nevidomskyy, V. Dmitriev, Angew. Chem. Int. Ed., **47**, 529 (2008).

[2] Y. Filinchuk, A.H. Nevidomskyy, D. Chernyshov, V. Dmitriev, Phys. Rev. B, **79**, 214111 (2009).

[3] I. Lindemann, R. Domènech Ferrer, L. Dunsch, Y. Filinchuk, R. Černý, H. Hagemann, V. D'Anna, L.M. Lawson Daku, L. Schultz, O. Gutfleisch, Chem. Eur. J., **16**, 8707 (2010).

P21: Update from the Swiss-Norwegian Beam Line BM01B at the European Synchrotron Radiation Facility

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Swiss-Norwegian Beam Lines, ESRF, Grenoble, France

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We present a short overview of experiments recently done on BM01B at the ESRF, which illustrate the broad range of techniques available on this beamline. In addition to the powder diffraction and EXAFS instrumentation, an on-line Raman spectrometer is now in routine use. Many experiments are profiting from the possibilities for rapid changeover from powder diffraction to EXAFS, facilitated by new X-ray optics which has been installed.

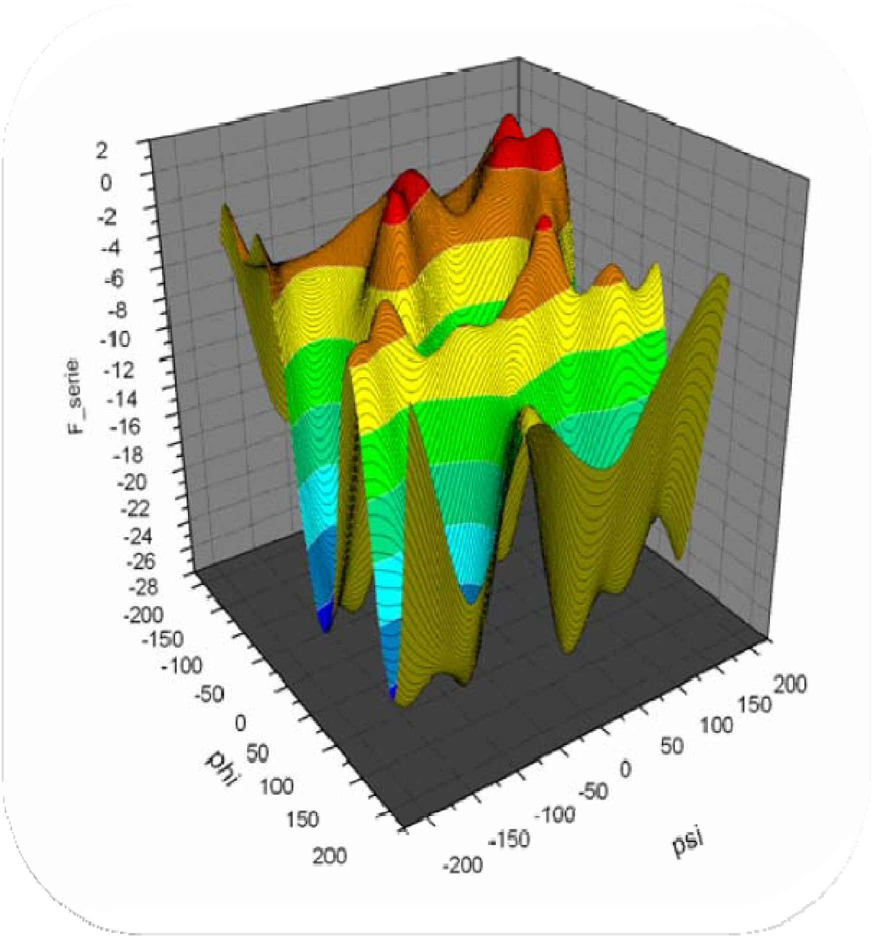
The wide and growing interest within our user community in catalysis has led to the development of in-situ techniques at SNBL. A fully automated gas mixing rig permits a wide variety of gas mixtures to be prepared and passed though the sample under different conditions of temperature and gas pressure while carrying out the synchrotron experiment. An on-line mass spectrometer is available for analyzing the products of the catalytic reaction.

The installation of three Mythen strip detector units (kindly provided to SNBL by PSI) will greatly increase the opportunities for rapid data collection, which are essential when following the kinetics of in-situ reactions. Fast data collection will also facilitate the future development of modulation techniques in combination with phase sensitive detection.

P22: Ramachandran Restrained Simulated Annealing Applied to Polypeptide Powder Diffraction Data

Sebastian Basso [a], Céline Besnard [b], Phil Pattison [a,c], Marc Schiltz [a]

[a] Ecole Polytechnique Fédérale de Lausanne(EPFL)
[b] Université de Genève (UNIGE),
[c] Swiss-Norwegian Beamlines (SNBL) at the ESRF



The determination of molecular structures *via* conventional X-ray crystallography methods is dependent on the capacity of materials to crystallize. Recently, interest has grown in the technique of powder diffraction, which makes use of the once-discarded microcrystalline precipitate. Moreover, powder diffraction has become a well recognized method in the field of structure refinement and molecular replacement for relatively extensive polypeptide molecules, namely small proteins. However, it was not until recent advances made in direct-space methods that structure solution using powder diffraction data was enabled for a wide range of organic materials, including polypeptides comprising of up to 4 amino acid residues. Direct-space methods consists of global optimization techniques such as Monte Carlo, simulated annealing and genetic algorithm. Through the use of test cases of tri-, tetra- and penta-peptides, the process of simulated annealing is attempted to be improved by applying Ramachandran plot restraints on the torsion angles defined as part of the molecule expressed as a rigid body. These restraints are formulated as a penalty function representing the Fourier series in two dimensions (phi and psi) of the Ramachandran plot (as illustrated in the image) for each torsion angle pair.

P23: Update from the Swiss-Norwegian Beam Line BM01A at the European Synchrotron Radiation Facility

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We present a short overview of experiments recently done at SNBL, which illustrate the broad range of experimental techniques available for the Swiss crystallographic community. Our examples include both powder and single crystal diffraction, structural studies as function of temperature, external pressure, laser irradiation, and diffuse scattering.

Special attention will be paid to 3D mapping of reciprocal space, the approach we use to measure diffuse scattering due to structural and thermal disorder. Various applications of this technique will be given, such as diffuse scattering from structural disorder in a Prussian Blue analogues, thermal diffuse scattering in zinc, diffuse scattering in a relaxor and ferroelectric perovskites, superstructure reflections in cobaltites and charge-density wave compounds.

We also present plans for the future development of the beam line and new possibilities that will be offered to our user community.

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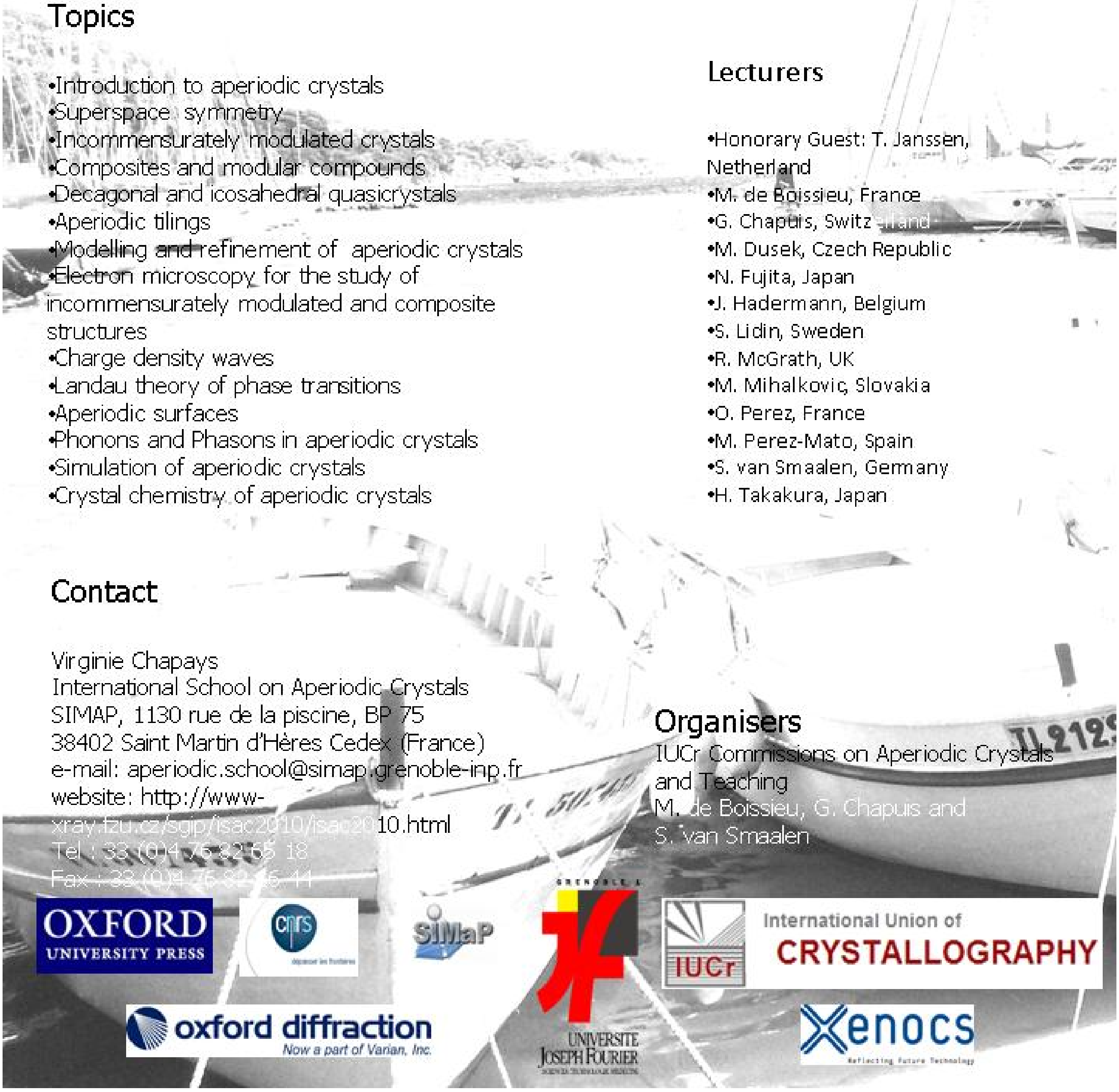

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Aug. 27-30	Darmstadt Germany	EPDIC-12 http://www.epdic12.org/	Expired
Aug. 27 – 28	Darmstadt Germany	ECM-26, Satellite Conference www.crystallography.fr/mathcryst/darmstadt2010.php	Expired
Aug. 29 – Sept. 3	Darmstadt Germany	ECM-26 http://ecm26.ecanews.org/	Expired
Sept. 16	Zürich CH	SCS Fall Meeting, Swiss Chemical Society http://www.scg.ch/x_data/event_pdf/FM10_Poster.pdf	Expired
Sept. 16, 20h	Geneva	Dinner of the SKG/SSCr Annual Meeting	Sept. 1, 2010
Sept. 17	Geneva CH	SGK/SSCr Annual Meeting http://diffraction.web.psi.ch/sgk-sscr-2010.htm	Expired
Sept. 26- Oct. 2	Carqueiranne France	International School on Aperiodic Crystals http://www-xray.fzu.cz/sgip/isac2010/isac2010.html	Expired
Nov. 22-27	Puebla Mexico	International Schools on Mathematical Crystallography http://www.crystallography.fr/mathcryst/ LatinAmericaSchools2010.php	to be announced
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2011

Jan. 17-25	Campinas Brazil	International School on 'New developments in the Field of Synchrotron Radiation'	to be announced
Jan. 23-28	Stoos CH	5th International Symposium on Hydrogen Storage www.empa.ch/h2e-symposium	Oct. 15, 2010
June 13-26	Zürich CH	The Zurich School of Crystallography: Bring Your Own Crystal, http://www.oci.uzh.ch/diversa/xtal_school/	see add inside
July 17-21	Prague Czech Rep	5th European Conference on Neutron Scattering http://www.ecns2011.org	to be announced
Aug. 22-29	Madrid Spain	IUCr-2011, 22nd General Assembly and Congress of IUCr http://www.iucr.org/iucr/cong/iucr-xxii	to be announced
Sept. 15-16	Villigen CH	JUM@P 11: Second Joint Users Meeting at PSI http://indico.psi.ch/conferenceDisplay.py?confId=42	to be announced
Sept 12-16 (tentatively)	Bern CH	SGK/SSCr Annual Meeting	to be announced

2012

Aug/Sept.	Bergen Norway	ECM-27	to be announced
April 29- May 4	Vancouver Canada	ARRS 2012 – Meeting of the American Roentgen Ray Society	to be announced
to be decided	Villigen CH	PSI Powder Diffraction Summer School	to be announced

2013

	Moscow Russia	17th International Zeolithe Conference	to be announced
April 14-19	Washington DC, USA	ARRS 2013 – Meeting of the American Roentgen Ray Society	to be announced
to be decided	Zürich CH	The Zurich School of Crystallography: Bring Your Own Crystal, http://www.oci.uzh.ch/diversa/xtal_school/	to be announced
to be decided		ECM-28	to be announced

2014

August	Montreal Canada	IUCr-2011, 23rrd General Assembly and Congress of IUCr http://www.iucr.org/iucr/cong/iucr-xxiii	to be announced
to be decided	Villigen CH	PSI Powder Diffraction Summer School	to be announced

Become a member of SGK/SSCr

If you are working in the field of crystallography, you will be interested to become a member of our society. For more information as well as online registration, please have a look on our website (http://www.sgk-sscr.ch). Presently, the yearly membership fee is sfr. 30 (sfr. 10 for students). For new members, the membership is free until the end of 2007. Please note: SGK/SSCr members can also apply to be a member of the subsection crystal growth (no additional charge) or for individual membership of the European Crystallographic Association, ECA (additional charge: 10 Euro).

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Highest degree received	
from university	
Present position	

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or use our online application form **at** http://www.sgk-sscr.ch

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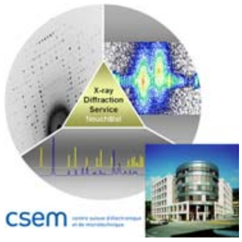

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