



Schweizerische Gesellschaft für Kristallographie
Société Suisse de Cristallographie
Società Svizzera di Cristallografia
Swiss Society for Crystallography

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

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Member of the
Swiss Academy of Sciences

SGK / SSCr NEWSLETTER

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Program of the SGK / SSCr annual assembly
to be held on Sept. 8, 2014 at EMPA Academy in Dübendorf,
including all abstracts submitted.

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EMPA-Akademie: a location for the
annual meeting of the SGK / SSCr (8. Sep. 2014)

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The President's Page

The 10th Ewald Prize has been awarded 2014 jointly to Prof. T. Janssen and Prof. A. Janner (both from the Institute for Theoretical Physics, University of Nijmegen, The Netherlands) for "*The development of superspace crystallography and its application to the analysis of aperiodic crystals*". Their investigations made a major input to crystallography by the extension of the space group classification to dimensions greater than 3. Nowadays, the superspace approach is integrated in many programs such as JANA. The mathematical concept



and the importance was nicely shown by professor Janssen in his opening lecture of the 23rd IUCr congress in Montreal. A recent review paper by Janssen & Janner, *Acta Cryst* **B70** (2014) 617-651 entitled "*Aperiodic crystals and superspace concepts*" has just been published. Our best congratulations to Prof. Aloysio Janner who is one of the "Gründungsmitglieder" of the SGK-SSCr, and has been continuously a full member of the society since 1969.

Another highlight was definitely the lecture of Nobel Prize Laureate Prof. D. Shechtman from Technion, Haifa, Israel. Not only has he outlined the history of his discovery, but also the skepticism he was facing, both in his own group and by the reviewers of his papers, as the discovery was in contradiction to generally accepted concepts in crystallography at this time. On the other hand, we were positively impressed on the manner IUCr supported his ideas, for example by redefining the crystal concept in a "*Report for the Executive Committee*", published in *Acta Cryst* **A48** (1992) 992-946, contributed on page 928 by the "*Ad Interim Commission on Aperiodic Crystals (Chair: J.M. Perez-Mato, members: W. Steurer, G. Chapuis, M. Farkas-Jahnke and M.L. Senechal)*".

Beside the many microsymposia with topics on the leading-edge of materials science, I specially would like to mention the keynote lecture from J. Rodriguez-Carvajal from ILL Grenoble entitled "*Sixty Five Years of Magnetic Structures: Present and Future of Magnetic Crystallography*" showing the enormous progress achieved in this field and the importance, crystallography will play in the future for the determination of magnetic properties.

My best wishes and hope to see many of you on Monday, Sept. 8, 2014 at our annual meeting at EMPA Dübendorf.

Jürg Schefer

News for and from members

We welcome the following new members of the SGK/SSCr

Mr. Matteo Brighi, University of Geneva
Ms. Jacinthe Gagnon, University of Fribourg
Ms. Milene Tan, University of Fribourg
Ms. Melanie Rohner, University of Zürich

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
- Applicant **MUST** be a member of our society

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

Travel grants are a good opportunity for young scientists to profit from our society during a period where they have low income. By subsequently becoming a long-term member of our society, you can return this good-will to the next generation.

Details for applications are given at:
<http://www.sgk-sscr.ch/TravelGrants.pdf>

Conference report: Powder Diffraction School of PSI, July 1 – 4, 2014

Contributed by Antonio Cervellino,

Instrument Scientist at the Materials Sciences Beamline of the SLS.

The 2014 edition of the Paul Scherrer Institute's Powder Diffraction School – entitled to “Modern Synchrotron Methods” - has been as all past editions a great success. The attendees were 53, that is the maximum number allowed by the allocated spaces; of those one half (27) were from Switzerland, from various institution (EMPA, Uni Bern, ETHZ, PSI, Uni Zurich, CERN, Dectris). The other half came mainly from the EU, with presences also from Serbia, Russia, Japan and South Africa. The program included three half-day lectures sessions, where different speakers have illustrated the many souls of powder diffraction methods, especially when performed at a synchrotron or a large-scale facility. Lectures on neutron powder diffraction and on powder-like diffraction at the SwissFEL have taken care of broadening the scope. Following that, three 4-hour experimental sessions at the Powder Station of the X04SA – Materials Science beamline of the Swiss Light Source at the PSI have been conducted, where the participants, subdivided into smaller groups, have had the opportunity to see how a successful synchrotron XRPD experiment should be carried out in detail (Fig.1, Dr. Nicola Casati in the PD hutch with a group of attendees). Model experiments on different samples have been performed, some of them aimed to a crystal structure solution, others aimed to total scattering analysis.





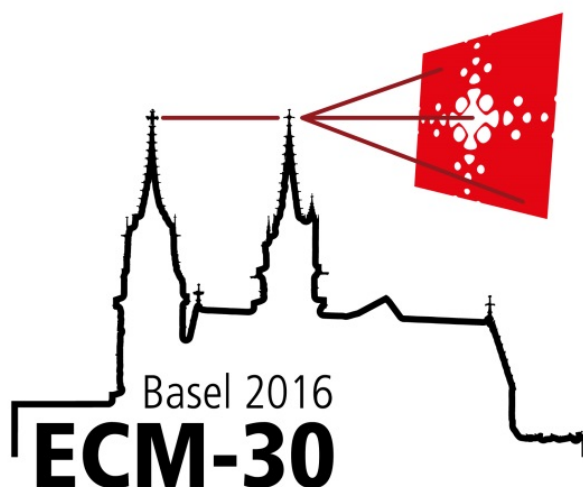
As the most important step in XRPD is the data analysis, six 4-hour demo sessions have been delivered, each illustrating one specific software package, including some of the most frequently used programs in the PD community: TOPAS, Fox, FullProf, PM2K, Debussy, Pdfgetx3. A nice social dinner – based on a rich Swiss barbecue at the Trotte Villigen – provided also some needed relax and increased the chances of useful professional contacts (Fig. 2, group picture in front of the Trotte Villigen, 2.7.2014).

The organizers (Dr. Antonio Cervellino and Dr. Nicola Casati, SLS, PSI; Mrs. Martina Füglistner, SLS secretary office) wish once more to thank the lecturers that, with their exciting lectures and their clear conduction of software sessions, have given the participants an enriching experience: Prof. Bruce Patterson, Uni ZH; Prof. Radovan Černý, Uni GE; Dr. Lynne McCusker, ETHZ; Dr. Steven Van Petegem, NUM, PSI; Dr. Matteo Leoni, Uni Trento, Italy; Dr. Ruggero Frison, CNR, Italy; Dr. Simona Galli, Uni Insubria, Italy; Dr. Fabia Gozzo, Excelsus Structural Solutions; Dr. Arnt Kern, Bruker Corp.; Dr. Dubravka Šišak-Jung, Dectris Ltd.; Dr. Denis Sheptyakov, NUM, PSI. Help from Dr. Martin Fisch, Uni Bern & PSI, has been invaluable. The organizers very gratefully acknowledge the support of the Swiss Crystallographic Society and of the industrial sponsors (Excelsus Structural Solutions, Dectris Ltd., Bruker Corp.) that have contributed to the success of this event.

Result of the Logo Competition for ECM-30 Basel, 2016

Six logos in 14 variations were submitted and presented to the local organizing committee of ECM-30 by E-mail for pre-selection. Out of the short list – made by a Doodle vote – the committee selected the logo designed by Fabienne Gschwind and Anthony Linden at its first meeting at the congress center in Basel, February 12, 2014, to be the official ECM-30 logo.

Aspects taken into consideration were not only the quality of the design, but also its suitability for use on small items, such as memory sticks, as well as on large presentation screens.



The selected logo skillfully combines the silhouette of Basel cathedral with the logo of the Swiss Society of Crystallography. The left spire of this monument in the city of Basel acts as a light source and the light impinges upon the cross on the pinnacle of the right spire. The Fourier transform of the cross results in the reciprocal space image that is our Society's logo. To integrate a mathematical relationship in the logo nicely appreciates the fact that important mathematicians such as Daniel Bernoulli (1700-1782), his nephew Jacob Bernoulli (1655-1705) and Leonard Euler (1707-1783) made important science contributions during their time in the host city of ECM-30.

The logo was presented to the ECA-councilors at a meeting held during the 23rd IUCr Congress in Montreal, August 9, 2014, and it will be on all our future ECM-30 presentations. Feel free to use it (www.sgk-sscr.ch)!

Congratulations and thanks to the authors of the selected logo and also to the vision of the original authors of the logo of our society, who were thinking well ahead of its use for ECM-30! Thanks also to all other contributors to this competition.

SGK / SSCr Finances

Summary of the SGK / SSCr Finances

	CHF
Total 01.12.2013	39'378.60
UBS account	21'575.64
CS account	18'123.69
Cash on hand	639.70
<hr/>	
Total 31.12.2013	40'339.03
Balance	960.43

SGK Financial Report 2013

UBS Account

CHF

UBS Balance 1.1.2013

20'708.38

Credits:

Membership dues	5'140.62
128x30.- (full member) 13x10.- (students) 9x130.- (companies)	

SANW reimbursement for Annual Meeting 2013	3'000.00
SANW for ECM delegate 2013	2'000.00
SANW funds for young scientists travel grants	1'500.00
SANW Promotion ECA meeting 2016	5'000.00

Interest	<u>21.60</u>
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Total Income

16'662.22

Debits:

Membership dues to SANW (204 members)	1'183.00
Promotion ECM 2016 (cards)	4'700.45
Promotion ECM 2016 (poster at ECM 2013)	145.00
Promotion ECM 2016 (calendar)	840.00
General expenses (switch)	17.00
Travel costs for ECM (Schaefer and Fromm)	2'000.00
Annual Meeting (MISSCA2013)	3'000.00
Travel Grants (Liuzzi, Cheremond)	1'500.00
Seminar Schetchman Fribourg	1'000.00
Support for Zurich school	1'025.40
Bank and Post expenses	384.11

Total Expenses

15'794.96

Income – Expenses

867.26

Starting Balance + Income – Expenses

21'575.64

UBS Balance 31.11.2013

21'575.64

Cash on Hand - 2013

	CHF
Status 1.1.2013	653.50
<u>Credits:</u>	
	<hr/>
Total Income	0.00
<u>Debits:</u>	
Post expenses	13.80
	<hr/>
Total Expenses	13.80
Balance (Income – Expenses)	-13.80
Starting Balance + Income – Expenses	639.70
Cash on Hand 1.1.2014	639.70

Credit Suisse (savings account)

	CHF
Status 01.01.13	18'016.72
Interest (1.125% or 1%, resp.)	106.97
Withholding Tax*	-.-
	<hr/>
Balance 31.12.13	18'123.69

*(only applicable when amount of interest exceeds CHF 200)

Revisorenbericht für die Jahresrechnung 2013 der Schweizerischen Gesellschaft für Kristallographie (SGK)

Konten:

UBS	UBS	279-C0291110.0
Credit Suisse	CS	913652-00

Die Unterzeichneten haben Kenntnis genommen von der Jahresrechnung der Schweizerischen Gesellschaft für Kristallographie. Die Rechnungsprüfung betrifft die Periode vom 1. Jan. 2013 bis 31. Dez. 2013. Die Unterzeichneten stellen fest, dass die Abrechnung mit den vorgelegten Belegen übereinstimmt.

Am 31. Dez. 2013 ist der Stand der Konten und der Kasse:

UBS	SFr.	21'575.64
CS	SFr.	18'123.69
Kasse	SFr.	639.70
Summe SGK	SFr.	40'339.03

Die Unterzeichneten beantragen der Versammlung die Entlastung des Kassiers und der Revisoren für die geprüfte Periode.

Ort / Datum
BERN, 27 März 2014

Unterschriften


B. Spingler
(Universität Zürich)


K. Schenk
(EPF Lausanne)

SGK Budget 2014 (Proposal)

<u>Credits:</u>	Budgeted
Membership dues (148-F, 9-J, 19-S)	5'000.00
SANW reimbursement for Annual Meeting 2014 *	2'500.00
SANW Poster prize annual meeting 2014 *	500.00
SANW reimbursement for IUCr delegate 2014 *	3'000.00
SANW young scientists travel grants *	1'500.00
Interest (est.)	100.00
Total Income	12'600.00

* SANW has not communicated yet the sponsorship for 2014

<u>Debits:</u>	
Membership dues to SANW (167 members)	1'200.00
Annual meeting	3'000.00
Travel Grant for IUCr delegate (2014)	3'000.00
Travel Grants to Young Scientists	1'500.00
SGK support for PSI School	2'000.00
Workshop at EPFL	1'000.00
ECA national membership dues 2014 (150€)	200.00
Expenses for ECM2016*	4'500.00
Bank charges	200.00
Total Expenses	16'600.00
Income – Expenses	-4000.00

* To be claimed from the budget of the ECM2016 meeting

**Swiss Crystallographic Association
SGK / SSCr
Annual Meeting 2014**

Abstracts and Program



Monday, September 8, 2014

Swiss Federal Laboratories for Material Science and Technology
Empa AKADEMIE
Ueberlandstr. 129
8600 Dübendorf

Meeting Title: **Modern Crystallography in Applied Research**

The 2014 annual meeting of the SGK/SSCr and general assembly will take place at the Swiss Federal Laboratories for Material Science and Technology Empa on Monday 8th of September 2014. Two invited speakers from academia, nine selected oral presentations and a poster session will show recent developments in the field. A visit of the new Center for X-ray Analytics at Empa will be organized.

More details on the website: <http://www.empa.ch/sgk>

Registration: Please register on our webpage www.empa.ch/sgk. The meeting is free of charge (except for accommodation).

Location:

Empa, Dübendorf

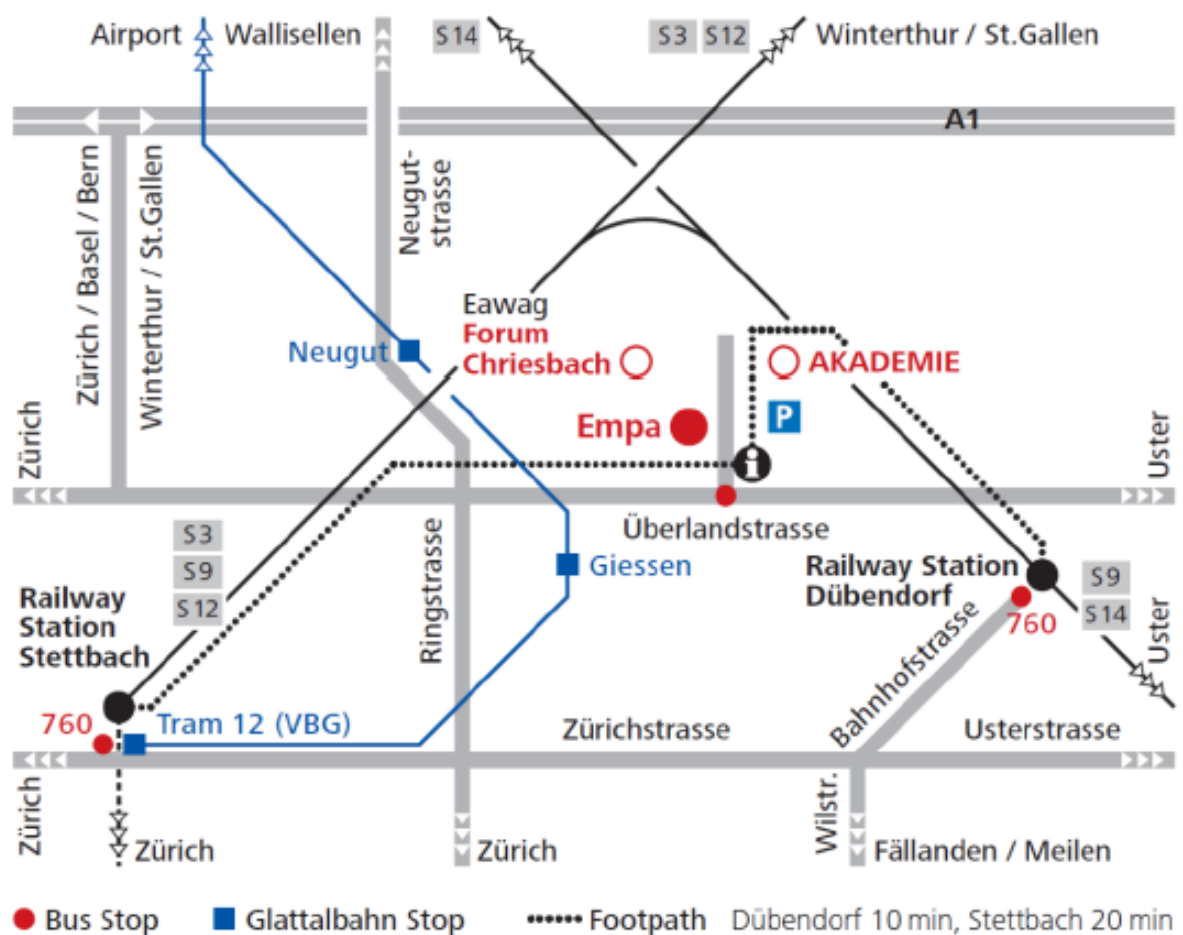
Überlandstrasse 129

AKADEMIE

GPS: Eduard Amstutz-Strasse

There is only limited parking available; please do use public transport.

Maps & Directions:



Program

9.00-10.00		Registration and Poster session
10.00-10.15		Welcome messages
10.15-11.00	IT1	Julian Stangl (Johannes Kepler University, Austria): "Crystallography at the Nanoscale: Structure and Strain in Semiconductor Nanowires"
11.00-11.15		Coffee break and Posters
		Session 1 (Inorganic Materials)
11.15-11.40	O1	Erik Stronks: "Modelling diffuse scattering of the disordered crystal structure of Na_2SiF_6 "
11.40-12.05	O2	Donat Adams: "Transition Mechanism of ferroelastic Phase Transitions in perovskites."
12.05-12.30	O3	Céline Besnard: "Structural investigations of the HS-LS relaxation dynamics on the porous coordination network $[\text{Fe}(\text{pz})\text{Pt}(\text{CN})_4]\cdot 2\text{H}_2\text{O}$ "
12.30-13.00		Lunch and Posters
13.00-13.45		General Assembly of the SGK
13.45-14.00		Coffee break and Posters
14.00-14.45	IT2	Arndt Remhof (Empa): "Structure and Dynamics of Novel Energy Materials"
14.45-15.10	O4	Aurélien Crochet: "Kinetics of Ion Transport through Supramolecular Channels in Single Crystals"
15.10-15.35	O5	Yolanda Sadikin: "Structural Analysis of Possible Lithium Migration Paths in Borohydrides"
15.35-16.00	O6	Xavier Maeder: "Cathodic Arc Evaporation of Oxide Coatings: Investigation of the Phase Transformation at the Target Surface and Deposition of Al and Hf oxides"
16.00-16.15		Coffee Break and Posters
16.15-16.30		Poster Prize Winners announcement
		Session 2 (Facilities and new instruments)
16.30-16.50	O7	Steven Peetermans: "How good is your sample? Revealing spatial variation in crystalline properties by means of energy-selective neutron imaging"
16.50-17.10	O8	Fiodar Kurdzesau : "Quantitative structure determination from energy dispersive Laue experiments"
17.10-17.30	O9	Luc Patthey: "The New Femto Second X-ray Laser Source at PSI"
17.30-19.00		Aperitif and Visit of the Center of X-ray Analytics

List of Posters

- P01 Z-DNA Crystal Structures Containing Cu(II)
- P02 Real space crystallography of a complex metallic alloy: high-angle annular dark-field scanning transmission electron microscopy of o-Al₄(Cr,Fe)
- P03 Single crystal diffraction in magnetic and electric fields
- P04 HEIMDAL: A time-of-flight neutron powder diffractometer at the European Spallation Neutron Source ESS for in-situ/in-operandi materials science studies
- P05 Pyridine Schiff-base ligands and their antimicrobial complexes
- P06 Relations between the 14 Bravais types of lattices
- P07 Phase transitions in borohydride perovskites
- P08 Studying structure disorder in DL-Norvaline by single crystal diffuse scattering
- P09 Membrane Supports for X-FEL Based Protein Crystallography
- P10 Transmission electron backscatter diffraction to characterize nanocrystalline electrodeposited materials
- P11 Stabilized ZrO₂: Ageing and Healing as seen by Powder X-ray Diffraction
- P12 Polymorphism, what it is and how to identify it
- P13 XRPD study of the photoinduced HS ® LS relaxation of the hydrated [Fe(pyrazine)Pt(CN)₄] polymeric network structure
- P14 Study on the different conformation of disulfide complexes
- P15 Polyanionic Cathode Materials for Sodium Ion Batteries
- P16 XRD characterization of III-V materials for CMOS technology
- P17 Multitopic Precursors for Oxide Materials' Synthesis
- P18 ZEBRA: The new neutron single-crystal neutron diffractometer optimized for small samples and extreme conditions at SINQ
- P19 The crystallization of melt-spun poly(3-hydroxybutyrate) (P3HB) fibers investigated by WAXD
- P20 Using synergy of experimental and computational techniques to solve monomer-trimer dilemma
- P21 Recent challenges for CMOS-Detector based Systems

Agenda of the SGK/SSCr General Assembly 2014

Academy of the EMPA Dübendorf, Monday, 8.9.2014, 13:00

The minutes of our last General Assembly (2013) in Como have been published in the SGK / SSCr Newsletter Nr. 89, which was distributed in December 2013, and is also available online at <http://www.sgk-sscr.ch/newsletter/>

- 1) Feststellung der Beschlussfähigkeit gemäss Art. 12/by-laws
- 2) Antrag auf Genehmigung der Minutes General Assembly 2013, Como
- 3) Jahresbericht
le rapport annuel
- 4) Jahresrechnung
les comptes annuels
- 5) Aufstellung des Budgets für das kommende Jahr/
le budget proposé pour l'année suivante
- 6) Festsetzung des jährlichen Mitgliederbeitrages/
le montant de la cotisation annuelle

Antrag des Vorstandes : wie bisher (CHF 30/regular und CHF 10/students)

- 7) Wahlen/Elections:
 - a) election / confirmation of the Board members
 - b) election / confirmation of the president
 - c) election / confirmation of the auditors
 - d) delegates to ECA and IUCR
- 8) Information on the progress of ECM-30 in 2016 (Basel)
- 9) Annual Meeting 2015: Location ?
- 10) Anträge von Mitgliedern
other motions of members

Meeting Sponsors



Zentrum für Wissenstransfer



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Akademie der Naturwissenschaften
Accademia di scienze naturali
Académie des sciences naturelles

Gold Sponsor

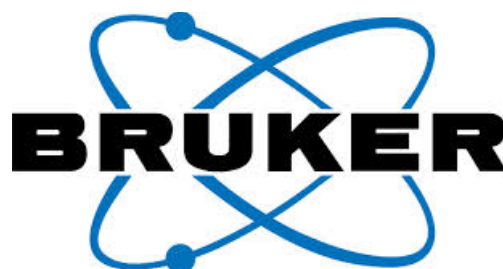


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Abstracts of invited talks and oral contributions

IT1:	Crystallography at the Nanoscale: Structure and Strain in Semiconductor Nanowires
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J. Stangl,¹ D. Kriegner,¹ M. Keplinger,¹ B. Mandl,^{1,2} R. Grifone,^{1,3} F. Bechstedt,⁴ C. Panse,⁴ V. Chamard,⁵ M. Borgström,² K. Deppert,² A. Diaz,⁶ T.U. Schüllli,³ G. Bauer¹

¹Johannes Kepler University Linz, Austria; ²Lund University, Sweden; ³ESRF Grenoble, France; ⁴Friedrich Schiller University Jena, Germany; ⁵University of Marseille, France; ⁶Paul Scherrer Institute Villigen, Switzerland

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Semiconductor nanowires attract scientific interest for several reasons. They are model systems for quantum confinement effects, but also promising as elements for solar cells or fast electronics, to name only a few. Especially in III-V semiconductors, an important issue is polytypism, leading to nanowires crystallizing in several crystal structures (see Fig. 1), which are not stable bulk phases [1,2]. This enables new types of heterostructures, switching only the crystal structure, but not the material. Also strain plays an important role in nanowires and can be used to modify properties. This talk reviews results on the crystallography of nanowires and the effects of strain, obtained using synchrotron radiation and focused x-ray beams.

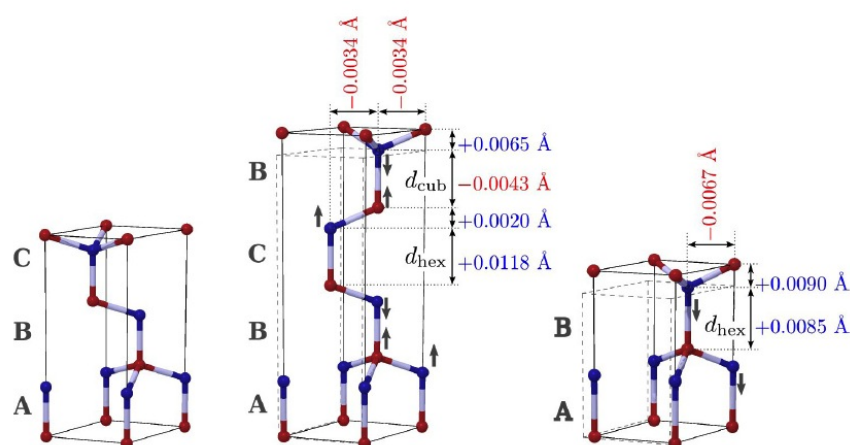


Figure 1: Crystal structures occurring in III-V semiconductor nanowires: cubic zincblende (left), hexagonal 4H (middle) and wurtzite (left). The depicted changes in bond lengths upon change from cubic to hexagonal structures are exaggerated.

References:

- [1] D. Kriegner, C. Panse, B. Mandl *et al.*, *Nano Letters* **2011**, 11, 1483–1489.
- [2] D. Kriegner, S. Assali, A. Belabbes *et al.*, *Phys. Rev. B* **2013**, 88, 115315.

Arndt Remhof

*Empa, Swiss Federal Laboratories for Science and Technology,
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The conversion and storage of energy is becoming increasingly important. Finite reserves of fossil energy carriers and growing environmental concerns are leading to an increased use of so-called renewable energy sources such as solar or wind energy. A future society therefore requires new materials to harvest energy fluxes and synthetic energy carriers. In contrast to the fossil energy carriers, the renewables deliver energy fluxes. Hydrogen would be an ideal energy carrier; its energy density exceeds that of gasoline by a factor of 2.5. At ambient conditions however, 1 kg of hydrogen requires a volume of 11 m³. Chemical hydrides reach volumetric hydrogen densities of more than 140 kg/l, exceeding the density of liquid hydrogen by a factor of 2. High volumetric densities combined with high gravimetric densities are realized in aluminum- and boron-based complex hydrides of the type M(AlH₄)_n and M(BH₄)_n, where M is a metal and n is the metal's valence. Because of its high hydrogen content of 18.5 wt %, lithium borohydride (LiBH₄) is one of the most discussed lightweight complex hydrides. It is also considered as a potential solid-state electrolyte for lithium-ion batteries and fuel cells, due to its high lithium ion conductivity of $\sigma = 1 \times 10^{-3}$ S/cm in the hexagonal phase at 393 K. Apart from being an ionic conductor, the hexagonal phase is associated with dynamical disorder of the [BH₄]⁻ anions, which undergo reorientational jumps in the terahertz range.

We present the synthesis of lightweight complex hydrides and their hydrogen release reactions. On the example of LiBH₄, we discuss the effect of imposed disorder either by halide additives or by nano-confinement as well as the relation between the orientational disorder and ion conductivity.

References:

- A Züttel, A. Remhof, A. Borgschulte, O. Friedrichs,
Phil. Trans. R. Soc. A **2010** 368, 3329–3342.
- M. Matsuo, A. Remhof, P. Martelli, R. Caputo et al.,
J. Am. Chem. Soc. **2009**, 131, 16389–16391.
- A. Remhof, Ph. Mauron, A. Züttel, J. P. Embs et al.,
J. Phys. Chem. C **2013**, 117, 3789–3798.
- J. Stingl, F. Zamponi, B. Freyer, M. Woerner, et al *Phys. Rev. Lett.* **2012** 109, 147402.
- Y. Yan, A. Remhof, S J. Huwang, H. W. Li et al,
Phys. Chem. Chem. Phys., **2012**, 14, 6514–6519.
- Y. Yan, A. Remhof, D. Rentsch, Y. S. Lee et al, *Chem. Commun.*, **2013**, 49, 5234.

Erik Stronks¹, Hans-Beat Bürgi^{1,2}, Anthony Linden¹, Loes Kroon-Batenburg³

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Many crystalline materials are not perfectly ordered, but are disordered to a smaller or larger extent. The properties of such materials are often related to the nature of the disorder. In diffraction experiments, disorder is observable as diffuse scattering. In routine X-ray structure determinations, only Bragg reflections are considered, leading to average crystal structure models. Methods to derive the average structure from the Bragg reflections are very well established, whereas diffuse scattering is rarely accounted for and mostly ignored. Our attempts at modeling the disordered structure of crystalline sodium fluorosilicate will be presented. Although the average crystal structure is known [1], there is some uncertainty about the true space group [2]. Na₂SiF₆ assumes a crystalline morphology that resembles that of ice and is therefore known as an ice-analog material [3]. In the average crystal structure (in space group *P*321), the asymmetric unit contains two ordered sodium cations (both sitting on a two-fold axis) and two disordered SiF₆⁻² anions (one sitting on a 3-fold axis and the other on a 32 site). Each anion can occupy two alternative sites in the unit cell, related by a non-crystallographic mirror plane at *z* = ¼. The occupation is mutually exclusive for both anions. Diffuse scattering can be observed as planes perpendicular to *l* in the *hnl* and *nk**l* precession images where these diffuse planes can be found at integer *l*. For *l* odd, the diffuse scattering is more intense. Also there are diffuse clouds of intensity around certain Bragg peaks. In the *hkn* planes, diffuse streaks are visible parallel to **a**^{*}, **b**^{*}, and **a**^{*} - **b**^{*}, while some Bragg peaks have diffuse clouds of intensity around them. The pattern of streaks and clouds evolves when going to higher order planes. In the figure below, the *hk*1 precession image is illustrated. The observed diffuse scattering features will be interpreted in terms of structural models obtained by Monte Carlo simulations.

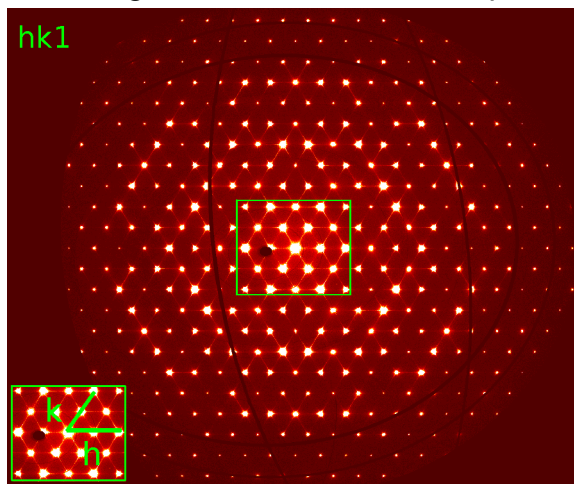


Figure 1: *hk*1 precession image.

References:

- [1] A. Zalkin, J. D. Forrester, D. H. Templeton, *Acta. Cryst.*, **1964**, 17, 1408
- [2] G.F. Schäfer, *Zeitschrift für Kristallography*, **1986**, 175, 269-276
- [3] H. Jeong, Y. Huh, *Cryst. Materials Letters*, **2010**, 64, 1816-1818

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We develop a formalism which allows to calculate the ionic contribution to the Free energy for materials which exhibit negative curvature of the potential energy surface with respect to atomic displacements. We show that the Free energy can stabilize crystal structures at temperature which appear dynamically unstable at $T = 0$. This formalism is computationally fast because it avoids statistical sampling through molecular dynamics calculations. The formalism is completely ab initio. It avoids statistical uncertainties and free model parameters but on the other hand gives insight into the mechanism of the structural phase transition.

We then apply this formalism to cryolite, i.e. the phase transition upon temperature from the $P2_1/n$ and the $Im\bar{m}$ space group. We calculate a phase transition temperature of 710 K, somewhat below the experimental value of 880 K. This can be related to the underestimation of the interaction of the vibrational states. We also calculate the main axes of the thermal ellipsoids and can to explain the increase of its volume for the fluorine by 200-300% throughout the phase transition with the calculated tunnelling states in the high temperature phase. Furthermore we show the relation between soft modes and degenerate eigenstates of symmetric double wells. We will give an outlook of the possible application of this to the problem of superconductivity, soft modes in other perovskites and the calculation of diffusion coefficients.

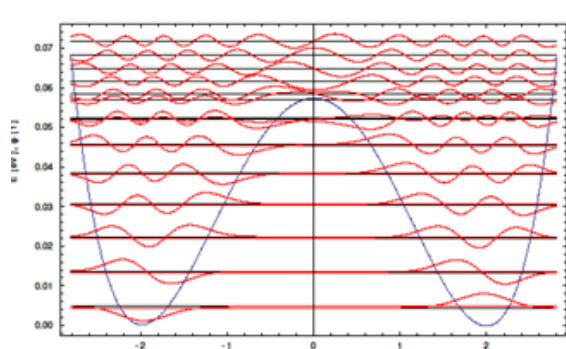


Figure 1: Ionic vibrational eigenstates in orthorhombic of cryolite (ionic vibration mode with lowest energy). The states are shifted to their corresponding energy and rescaled in order to enhance their visibility. The lowest states are degenerate and correspond to the well known soft modes.

References:

- H. Yang, S. Ghose, and D. Hatch, Phys. Chem. Minerals **1993**, 19, 528.
I. Errea, M. Calandra, and F. Mauri, Phys. Rev. B **2014**, 89, 064302.

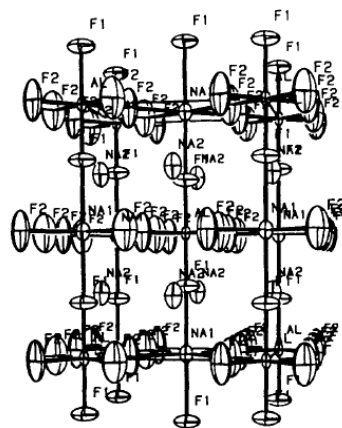


Figure 2: Thermal ellipsoids in the orthorhombic phase of cryolite at 890 K from Yang (1993). The elongated ellipsoids can be explained with tunneling eigenstates depicted in Fig. 1.

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Spin Crossover (SCO) Compounds can be converted between Low Spin (LS) and High Spin (HS) states under different external stimuli such as pressure, temperature, light or guest absorption. Interactions between the SCO centers give rise to the so-called cooperative effects, which governs the SCO behavior. The investigation of the HS-LS photo-excitation dynamics is of particular importance to understand the physics associated with cooperative effects. Crystallographic measurements are a very interesting tool in this field, since they provide direct structural information¹. Using synchrotron radiation, fast powder diffraction measurements were used to study the relaxation of the HS photo-induced state of the Hofmann-like network [Fe(pz)Pt(CN)₄]₂H₂O and revealed a complex relaxation behaviour mixing random HS to LS conversion and nucleation growth process.

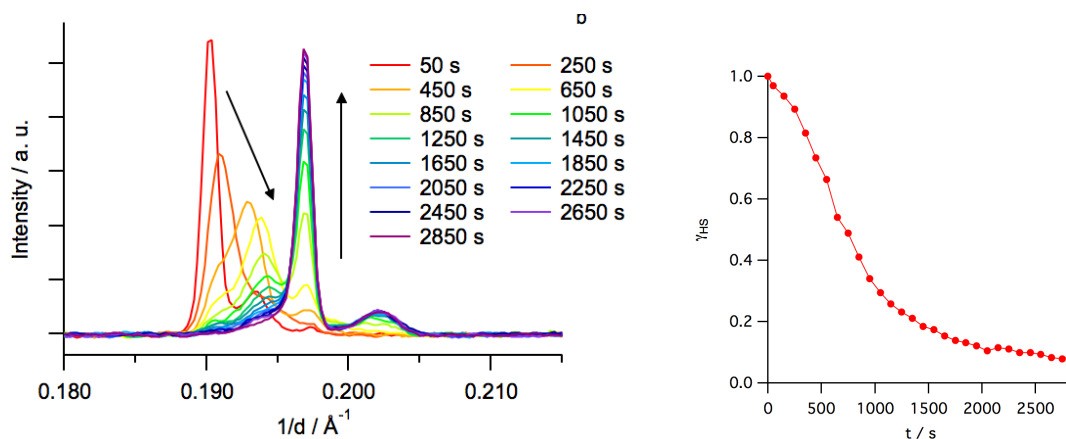


Figure 1: Time evolution of the low angle part of the Powder X-Ray diffraction pattern of [Fe(pz)Pt(CN)₄]₂H₂O at 10 K after irradiation at 532 nm (left) and relaxation curve showing the HS fraction as a function of time extracted from the powder data (right).

Reference:

1 Guionneau P., *Dalton Trans.*, **2014**, 43, 382

O4:	Kinetics of Ion Transport through Supramolecular Channels in Single Crystals
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Reactions in single crystals of supramolecular assemblies are most often associated with the breaking of the crystals into polycrystalline or amorphous materials, induced by the major transformations and related strain during the reaction. The rare examples in which the single crystallinity is maintained in supramolecular compounds during the reaction are based on the neutral sorption and desorption of guest molecules in robust three-dimensional (3D) metal–organic frameworks (MOFs) or [2+2] photodimerization processes in organic compounds or coordination polymers. Ion-exchange reactions, as reported, for example, for zeolites and polycrystalline MOFs, are often incomplete and determined by methods such as atomic absorption spectroscopy and energy dispersive X-ray spectroscopy. While 3D MOFs have been compared to zeolites, we now show the generation of one-dimensional (1D) channel systems based on crown ether ligands which perform alkali cation transport in analogy to ion channels in biology and synthetic organic channels in membranes. Crown ether complexes with alkali metal ions have been described, yet the number of compounds in which this kind of ligand assembles into 1D stacks is extremely limited.[7] In the current study we used trihalide anions of the type X_3^- , X_2Y^- , and XY_2^- ($X=I$, $Y=Br$) as structural pillars, which induce the stacking of dibenzo[18]crown-6 molecules. Single crystals of the resulting channel systems turned out to allow fast alkali cation exchange. To our knowledge, this is the first time that the activation energy for cation transport could be measured in supramolecular stacks. The crystals are very stable during these experiments as they remain unchanged in form and size.

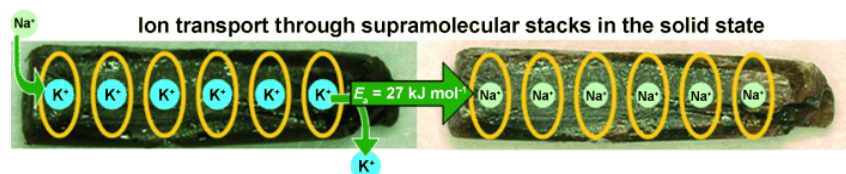


Figure 1: Single-Crystal to Single-Crystal transformation

Reference:

Cyrille D. Assouma, Aurélien Crochet, Yvens Chérémond, Bernd Giese and Katharina M. Fromm, *Angew. Chem. Int. Ed.* **2013**, 52, 4682 –4685.

O5:	Structural Analysis of Possible Lithium Migration Paths in Borohydrides
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Borohydrides have been attracting significant interests as potential solid electrolytes since the discovery of fast Li-ionic conduction in hexagonal LiBH_4 [1]. In general, borohydrides exhibit rich crystal chemistry (ionic crystals and frameworks containing mixed cations and mixed anions) which can be linked analogically to oxide family. This allows extensive possibilities of synthesizing new compounds in search for novel fast ion conductors with improved properties. Therefore, the understanding of the ionic migration path is important to screen ionic conduction through a wide range of new borohydride structures.

Studies of voids and channels as Li-ion migration paths in Li-containing borohydrides with different compositions will be the focus of this presentation. TOPOS software is employed to analyze geometrically the possible conduction space by using Voronoi-Dirichlet Polyhedra (VDP) method. Previously, this method has been systematically applied for oxide compounds [2]. In addition, migration paths can also be modelled as the path of lowest electron density [3] calculated by Hirshfeld surface analysis tool available in Crystal Explorer software. One example of the compounds studied, the high temperature LiBH_4 with hexagonal structure can be shown to possess 3D Li-ion conduction (Figure 1).

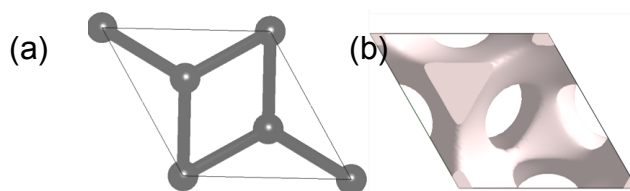


Figure 1: Possible Li^+ pathways in hexagonal LiBH_4 calculated from (a) VDP (b) Hirshfeld surface analysis

References:

- [1] M. Matsuo, Y. Nakamori, S. Orimo, H. Maekawa, H. Takamura, *Appl. Phys. Lett.* **2007**, 91, 224103(1)-(3).
- [2] N.A. Anurova, V.A. Blatov, G.D. Ilyushin, O.A. Blatova, A.K. Ivanov-Schitz, L.N. Dem'yanets, *Solid State Ionics.* **2008**, 179, 2248-2254.
- [3] M.Ø. Filø, M.J. Turner, G.V. Gibbs, S. Adams, M.A. Spackman, B.B. Iversen, *Chem. Eur. J.* **2013**, 19, 15535-155

O6:	Cathodic Arc Evaporation of Oxide Coatings: Investigation of the Phase Transformation at the Target Surface and Deposition of Al and Hf oxides
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Reactive cathodic arc evaporation is an attractive method to produce oxide coatings. One reason for this is the possibility to control the phases and phase compositions of the synthesized oxides by the chemical composition of compound targets utilized as cathodes for the evaporation. The oxide phases depend, however, also from other deposition conditions, e.g. the oxygen partial pressure. Oxygen influences the evaporation rates of the cathode material as well as the conditions under which the metallic vapour condenses and reacts at the substrate surface. In this context, Al-Hf composite targets have been investigated with the atomic compositions of: Al₇₅Hf₂₅, Al₇₀Hf₃₀ und Al₆₇Hf₃₃. The narrow composition range was selected to study the sensitivity of the surface modifications with respect to the target composition. The targets were operated with and without oxygen reactive gas. The phase composition at the target surface was determined by X-ray diffraction (XRD) analyses and compared for the different process conditions (Maeder et al. 2013). Coatings were deposited for each process parameter set utilized for surface treatment of the targets. The compositions of the layers were determined by Rutherford backscattering spectrometry. XRD was used to determine the metallic and oxide phases in the layers. A comparison of the phase composition between layers and target surface is given and the possible processes during target surface modifications are discussed. Oxide coatings are well suited for applications in high temperature environment, especially as oxidation and diffusion barriers. In this context, high temperature XRD measurements up to 1300°C were also done to study the process of oxide formation from intermetallics in the layers (Maeder et al. in press).

References:

X. Maeder, M. Döbeli, A. Dommann, A. Neels, P. Polcik, R. Rachbauer, H. Rudigier, B. Widrig, J. Ramm. *Proceeding of the 18th Plansee Seminar, L.S. Sigl, H. Kestler and J. Wagner (Eds.), PLANSEE SE, Tyrol/Austria, 2013*, HM45, 1381.

X. Maeder, M. Döbeli, A. Dommann, A. Neels, H. Rudigier, B. Widrig, J. Ramm. *Journal of Surface Coating and Technology*, in press.

O7:**How good is your sample? Revealing spatial variation in crystalline properties by means of energy-selective neutron imaging**

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Common neutron imaging techniques study the attenuation of a neutron beam through a sample of interest using large ($\sim 10 \times 10 \text{ cm}^2$) position sensitive detectors of high spatial resolution ($< 100 \mu\text{m}$). The recorded radiograph show contrast depending on traversed material composition and thickness. It is routinely performed at PSI's neutron imaging facilities solving problems ranging e.g. from water management in fuel cells to virtually separating fossils from stone.

Imaging with a monochromator, one adds crystallographic properties to the list of observables, which can be understood in the context of the Bragg law $2d_{hkl}\sin(\theta_{hkl}) = \lambda$. Different types exist (mechanical velocity selector, double crystal monochromators) yielding different wavelength bandwidths.

In polycrystalline samples, sharp Bragg edges are formed as coherent elastic scattering at the hkl plane can occur for all wavelengths up to $2d_{hkl}$, after which a sharp increase in transmission intensity is observed. Their shape and position are a.o. influenced by texture, strain and phase.

In single crystals, all orientations θ w.r.t. the beam are no longer present and rather than Bragg edges the transmission spectrum shows discrete dips at wavelengths that fulfill Bragg's law. Their width and position reveal crystal mosaicity and orientation.

Examples are shown for microstructural changes across SwissFEL copper disks and variations in orientation and mosaicity in pyrolytic graphite monochromator crystals for the CAMEA neutron spectrometer currently designed at PSI (figure 1).

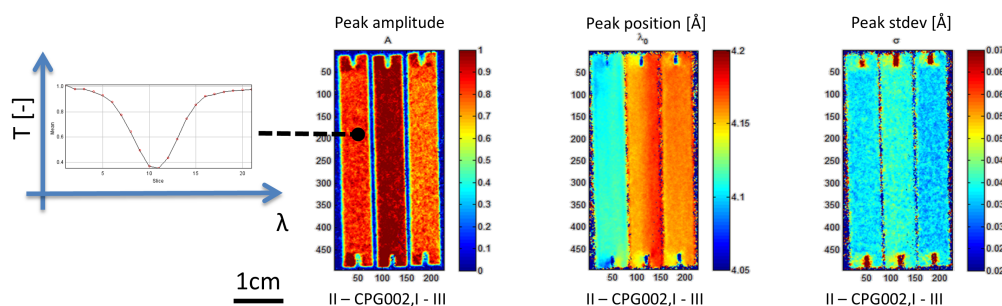


Figure 1. Pixelwise analysis of the transmission spectrum of three PG monochromator crystals and associated crystal properties (reflectivity, orientation and mosaicity)

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The use of 2D position and energy sensitive hybrid pixel Pilatus detectors enables energy-dispersive analysis of Bragg reflections [1]. This improves classical Laue experiments and allows quantitative crystal structure determination without having any priory information about the sample [2].

The energy-dispersive Laue diffraction (EDLD) experiments can be performed with a white beam from conventional X-ray tubes. It provides the better control of irradiation spectra by applying different voltage/current settings during the tube operation and allows combining the primary beam (**Figure 1(a)**) and XRD measurements using the same Pilatus detector.

In the present work EDLD experiments were done with MoK α X-ray tube using a 300K Pilatus detector (4-25 keV operation range). All data processing steps (finding the Bragg reflection energy and integrated intensity, spectral corrections from primary beam measurements, the sample absorption etc.) were done following in-house algorithms. Applying the developed intensity correction protocols the structure refinement of a reference quartz sample could be done with R-factor <0.10 (**Figure 1(b)**). Possible applications of this method (e.g. quantitative XRD studies with stationary crystals) and the details for its further development will be discussed.

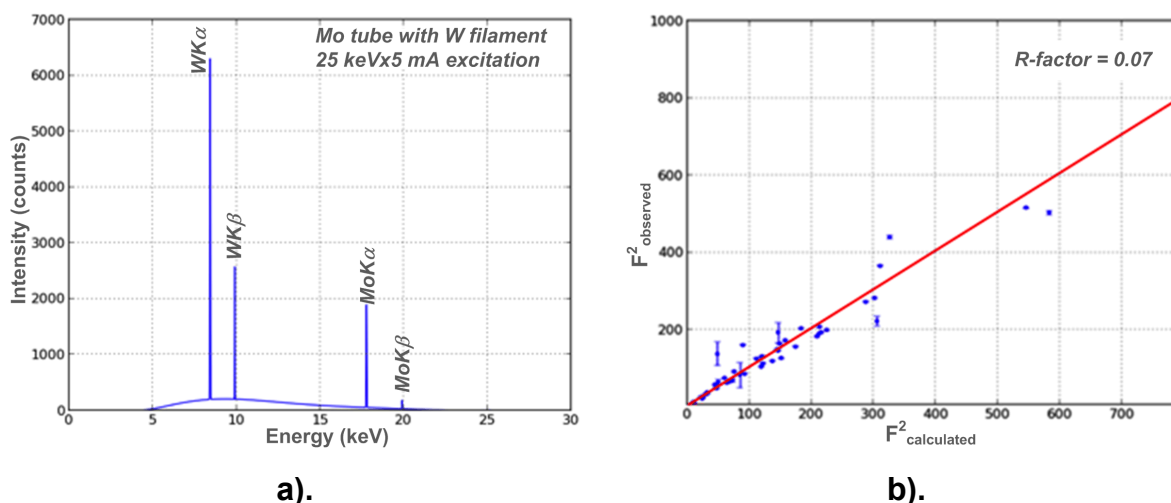


Figure 1: Primary beam measurements (a) and structure refinement (b) in EDLD experiments.

References:

- [1] P. Kraft, A. Bergamaschi, Ch. Brönnimann et al, *IEEE Trans.Nucl.Sci.*, **2009**, 56(3), 758-764
- [2] S. Send, M. von Kozierowski, T. Panzner et al, *J.Appl.Cryst.*, **2009**, 42, 1139-1146

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Paul Scherrer Institute is building an X-Ray Free Electron Laser (SwissFEL) facility, which will be in operation in 2017 and produce 20 fsec pulses of coherent x-rays in the wavelength range 0.1 to 7 nm, with extremely high peak brightness. These characteristics will provide opportunities for new experiments in chemistry, solid state physics, biochemistry and materials science. After a brief status report, the presentation will focus on novel applications, the description of the fundamental aspects of the planned facility with an emphasis on the photonics part of the project.



Figure 1: Air view of the SwissFEL building

References:

www.swissfel.ch

Abstracts of Posters

P01:

Z-DNA Crystal Structures Containing Cu(II)

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Z-DNA is a very promising concerning transcription regulation element and hence might be relevant for future cancer therapy. Still only few research groups investigated the functionality and structural stability of Z-DNA in the last years [1, 2]. The transition from B- to Z-DNA of poly d(GC) oligonucleotides can also be induced non-biologically by a high concentration of multivalent cations [3, 4, 5].

To study the interaction of such multivalent cations with the Z-DNA and their mode of stabilization [6], crystals of d(CG)₃ with copper(II) were grown. Three different crystal data sets could be collected, but so far only two of them could be solved. In this work we present the successful structure solution of the third crystal data set with modern solution strategies and the found interaction of copper(II) with Z-DNA in all three structures.

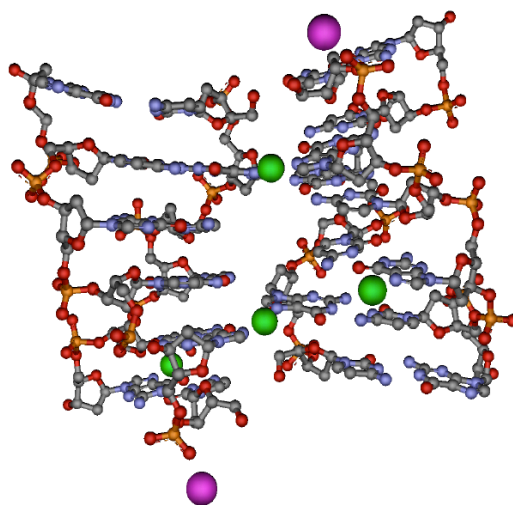


Figure 1: Representative structure of one of the three DNA crystals. Copper ions are shown in green and calcium ions in violet. Water molecules are omitted for reasons of clarity.

References:

- [1] A. Rich, S. Zhang, *Nat. Rev. Gen.* **2003**, 4, 566–572.
 - [2] J. Zhao, A. Bacolla, G. Wang, K. M. Vasquez, *Cell. Mol. Life Sci.* **2010**, 67, 43–62.
 - [3] F. M. Pohl, T. M. Jovin, *J. Mol. Biol.* **1972**, 67, 375–396.
 - [4] T. Biver, *Coord. Chem. Rev.* **2013**, 257, 2765–2783.
 - [5] T. F. Kagawa, B. H. Geierstanger, A. H.-J. Wang, P. S. Ho
J. Biol. Chem. **1991**, 266, 20175–20184.
 - [6] A. Medina-Molner, B. Spingler *Chem. Commun.* **2012**, 48, 1961–1963.
- SGK / SSCr Newsletter No.91 (2014)

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Al-Cr-Fe Complex Metallic Alloys (CMA) are corrosion resistant Al- based intermetallic phases (quasicrystalline approximants) stable in pHs ranging from 0 to 14 that can be exploited in resistant “multifunctional” coatings of Al alloys and steels [1].

High-angle annular dark-field scanning transmission electron microscopy (HAADF STEM) has been performed along the low-index zone axes of the α -Al₄(Cr,Fe) complex metallic alloy to obtain a real-space representation of the crystal structure and to elucidate the material's inherent structural disorder [2]. By comparing experiments with multislice STEM simulations, the model previously suggested by X-ray diffraction is further refined to provide a new set of positions and occupancies for the transition metal atoms. *Pmnb* is suggested as the new space group for the α -Al₄(Cr,Fe) phase. A nonperiodic layer-type modulation, averaged out in bulk diffraction methods, is detected, corroborating the need for complementing bulk diffraction analysis with real-space imaging to derive the true crystal structure of Al₄(Cr,Fe).

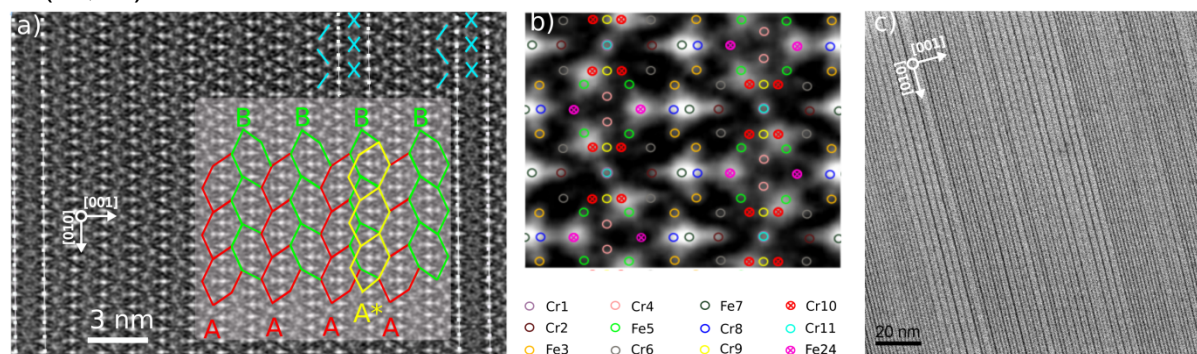


Figure 1 a) Noise- filtered STEM image viewed along the [100] zone axis. b) Superposition of the model on the experimental image. c) Bright field STEM image.

References:

[1] A. Beni, N. Ott, M. Pawelkiewicz, M. Wardé, K. Young, B. Bauer, P. Rajput, B. Detlefs, J. Zegenhagen, R. McGrath, M.-G. Barthés-Labrousse, L.P.H. Jeurgens, P. Schmutz, *Electrochemistry Communications*, 46 (2014) 13-17.

[2] R. Gaspari, R. Erni, Y. Arroyo, M. Parlinska-Wojtan, J. Dshemuchadse, C.A. Pignedoli, D. Passerone, P. Schmutz, A. Beni, *J. Appl. Cryst.*, 47 (2014) 1026-1031.

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Multiferroic materials have become of interest for their unusual low-temperature properties in general, and the tunability of the magnetic structure through an electric field and the electric polarization through a magnetic field in particular. The most promising candidates for such controllable multiferroics have been found among the materials with inherent geometric magnetic frustration.

Among these, the delafossite CuCrO_2 , which crystallizes in the rhombohedral $R\bar{3}m$ space group, is a multiferroic compound with an apparent strong coupling of spin and charge [1]. In contrast to other multiferroic compounds CuCrO_2 shows a spontaneous electric polarization upon antiferromagnetic ordering without an accompanying structural phase transition, thus the spiral magnetic ordering alone breaks the inversion symmetry. The peculiar magnetic structure of CuCrO_2 allows the direct quantitative analysis of the domain population [2].

In our contribution, we present a detailed study on CuCrO_2 single crystals using neutron diffraction in applied electric and magnetic fields. With the fields we were able to tune the multiferroic states in CuCrO_2 and could directly relate them to the underlying domain physics [3-5]. Surprisingly, the domain population is changed only slightly by the electric field and the observed multiferroic properties arise therefore only from a fraction of the whole crystal. Further, the sign reversal of the electric polarization through a reversed electric field is not accompanied by a domain redistribution. This indicates a coupling of the electric polarization to the chirality of the magnetic spiral. We will also present evidence that the three domain state is the ground state and that the one- or two-domain state is a non-equilibrium state. The underlying mechanism is speculative, but its existence is important to understand the multiferroic properties of CuCrO_2 .

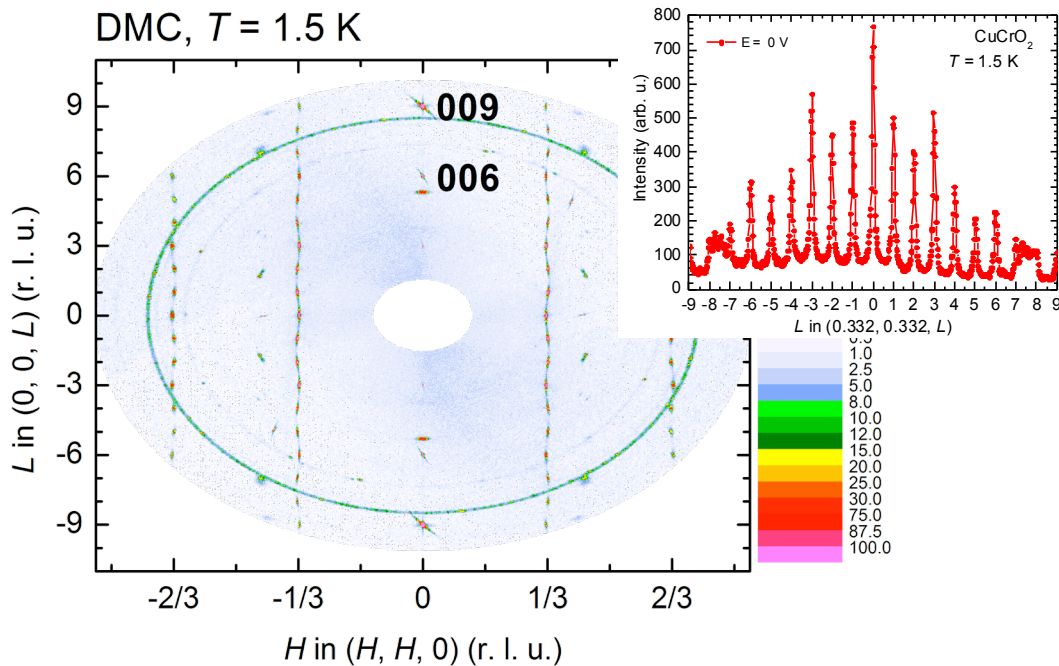


Figure 1: Reciprocal HHL-plane of CuCrO₂ at 1.5 K. The magnetic peaks are close to the $H = 1/3, 2/3$ positions, the nuclear 006 and 009 reflection are marked. The inset shows a section along L for $H = 1/3$.

References:

- [1] K. Kimura, H. Nakamura, K. Ohgushi, T. Kimura, PRB **78**, (2008) 140401
- [2] M. Frontzek, G. Ehlers, A. Podlesnyak, H. Cao, M. Matsuda, O. Zaharko, N. Aliouane, S. Barilo S. V. Shiryayev, J. Phys.: Condens. Matter **24** (2012) 016004
- [3] E. Mun, M. Frontzek, A. Podlesnyak, G. Ehlers, S. Barilo, S. V. Shiryayev, V. S. Zapf, Phys. Rev. B **89** (2014) 054411
- [4] G. Ehlers, A. A. Podlesnyak, M. Frontzek, R. S. Freitas, L. Ghivelder, J. S. Gardner, S. V. Shiryayev, S. Barilo, J. Phys.: Condens. Matter **25** (2013) 496009
- [5] M. Frontzek, J. T. Haraldsen, A. Podlesnyak, M. Matsuda, A. D. Christianson, R. S. Fishman, A. S. Sefat, Y. Qiu, J. R. D. Copley, S. Barilo, S. V. Shiryayev, G. Ehlers, Phys. Rev. B **84** (2011) 094448

P04:	HEIMDAL: A time-of-flight neutron powder diffractometer at the European Spallation Neutron Source ESS for <i>in-situ/in-operandi</i> materials science studies
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Developing new materials is of paramount importance to combat future energy demands, and environmental damage. Improvements in material performances are reached for example by the incorporation of advanced ceramics and polymers into heterogeneous systems. Their performances usually depend on the interplay between properties defined by the atomic, nano/mesoscopic and microscopic structure. *In-situ* and *in-operandi* investigations will be in the focus of such investigations. The instrument HEIMDAL proposed for the European spallation neutron source ESS will offer here perfect prospects, as the instrumental resolution of this powder diffractometer can widely be adapted and take full advantage of the broad pulse of ESS (2.86ms) offering highest intensity, or using a fraction of the pulse for highest resolution as shown in Fig. 1, giving gains factors of 3 to 290 compared to GEM (Tab.I). A thermal and a cold guides pointing to the same virtual source extend the spatial window of the instrument from an atomic scale ($0.3\text{\AA}^{-1} \leq Q \leq 50\text{\AA}^{-1}$) to a nano/meso scale, $0.002\text{\AA}^{-1} \leq Q \leq 0.1\text{\AA}^{-1}$ by adding a narrow-band SANS instrument behind. Our chopper system allows switching the different operation modes electronically. Traditionally such structural information has been collected in separated experiments such as powder diffraction (PD), wide angle diffraction scale, small angle diffraction and direct space imaging techniques (sub-micronic to millimeter scale). HEIMDAL can offer these options in at the same time, therefore for absolutely identical experimental conditions.

The top-loading geometry foreseen not only accepts auxiliary from the ESS pool (cryogenics, pressure cells, magnets) but also allows implementing bulky brought-in user equipment. It can be pretested off-line at the instrument, but already fully connected to the HEIMDAL electronics.

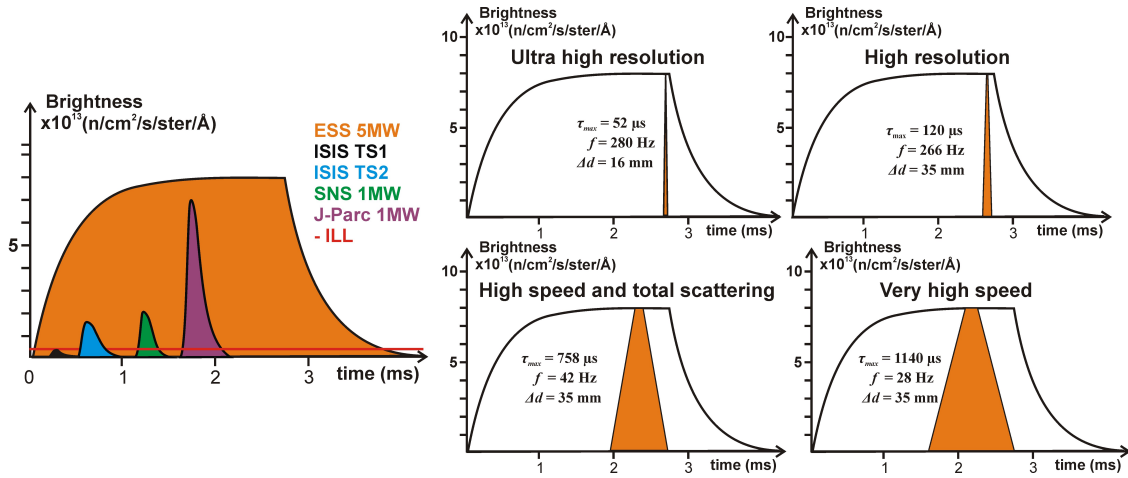


Figure 1: Comparison of pulse brightness at different facilities (far left), and the pulse used for obtaining ultra high resolution, high resolution, high speed and very high speed with HEIMDAL@ESS.

Table I: Instrument comparison, the different values for flux etc. are found at web pages and papers are more through description of the individual instruments can be found in appendix B. The $G_{eff} = \Phi D_{area} D_{eff} / (\Delta d/d @ 90^\circ)$, the D_{eff} is set to 100% for ³He and 60% for scintillation counters and ¹⁰B.

Instrument	Type	$\Delta d/d @ 90^\circ$	$\Delta \lambda$ (Å)	λ_{mean} (Å)	Q_{range} (Å ⁻¹)	flux (n/s/cm ²)	G_{eff}
GEM @ ISIS	TOF	0.50%	3.5	1.8	0.04-100	2e6	1
New Polaris @ ISIS	TOF	0.51%	5.5	2.9	0.7-125	~1e7	~7
Nomad @ SNS	TOF	0.60%	3.0	1.6	0.5-125	~1e8	~71(150)
PowGen @ SNS	TOF	0.50%	2	1.1	3-120	~2.5e7	~14
I-Materia@JPARC	TOF	0.50%	6	3.3	0.007-70	~1e8	~86
Nova@JPARC	TOF	0.50%	7	3.6	0.4-100	~4e8	~385
D20@ILL	CW	1.6%	-	1.3	0.2-8	~1e8	~2
Powtex@FRM2	TOF	0.60%	1.4	1.6	0.4-13	~1e7	~7
Wombat@OPAL	CW	1.0%	-	2.4	0.4-4	~1.3e8	~8

P05:	Pyridine <i>Schiff</i>-base ligands and their antimicrobial complexes
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Since decades, *Schiff* base complexes were widely studied because of their various catalytic properties^[1] and also their biological activity^[2]. We are presenting different pyridine based *Schiff*-base ligands, their reduced derivatives and complexes with copper(II) and silver(I). Introducing these two metals^[3,4] can impart or enhance antimicrobial properties of the synthesized ligands.

Symmetric pyridine based *Schiff*-base ligands, like the ones with an alkyl linker shown in *Figure 1*, are easy to synthesize in high yields and offer several coordination sites for diverse metal ions. Independent of the length of the alkyl chain most ligands crystallize in a monoclinic crystal system with a unit cell of $P2_1/c$.

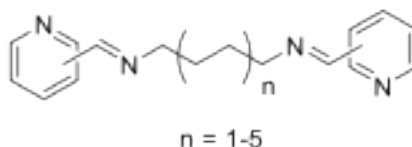


Figure 1: General scheme for symmetric alkyl linked pyridine based *Schiff* bases.

Adding, for example, copper(II) bromide to a ligand solution, an antimicrobial 2D coordination polymer of copper(II) bromide chains cross-linked by pyridine based *Schiff* bases is formed and can be further investigated for its application in medicine.

References:

- [1] K. C. Gupta, A. K. Sutar, *Coord. Chem. Rev.* **2008**, 252, 1420.
- [2] J. McGinley et al.; *Polyhedron* **2013**, 55, 169.
- [3] J. O’Gorman, H. Humphreys, *Journal of Hospital Infection* **2012**, 81, 217.
- [4] S. Eckhard, P. S. Brunetto, J. Gagnon, M. Priebe, B. Giese, K. M. Fromm, *Chem. Rev.* **2013**, 113, 4708.

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The *International Tables for Crystallography (ITC)* do not state explicitly, which of the 14 Bravais types of lattices are special cases of others, although they contain the information necessary to derive the result in two ways, considering either the symmetry or metric properties of the lattices. The first way was opened by C. Hermann in 1935, the second was followed by M. Klemm in 1982. It would be useful to have the result presented in the *ITC* in a way similar to the presentation of the subgroup relations between the 32 crystallographic point groups.

Order of point group

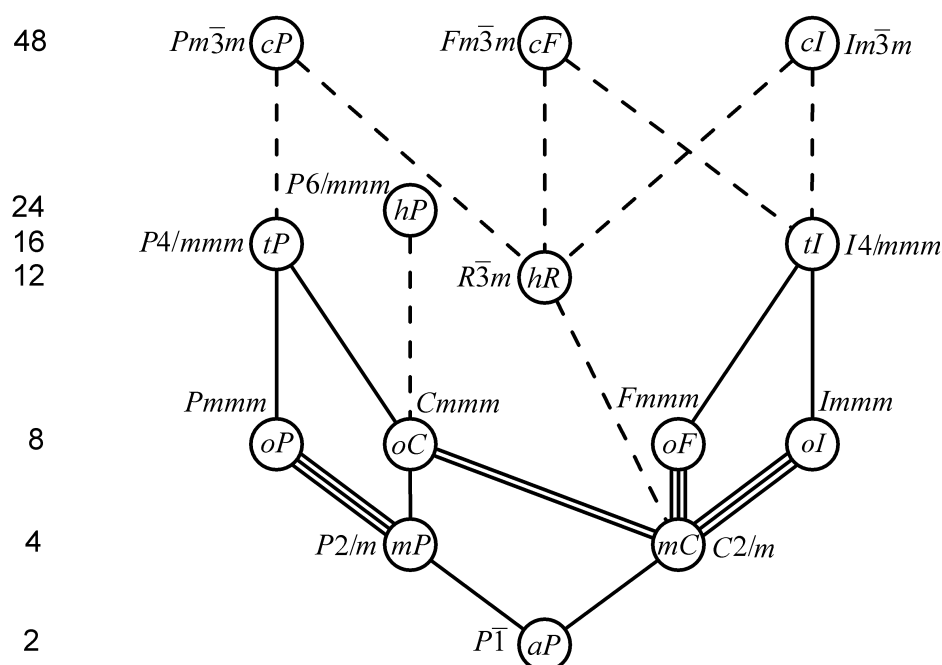


Figure 1 The Bravais type of the three-dimensional lattice at the upper end of a line is a special case of the type at its lower end. This follows from the subgroup relations among the space groups corresponding to the lattices: the group at the lower end of a line is a maximal *translationengleiche* subgroup of the group at the upper end. Solid lines indicate normal subgroups, dashed lines sets of conjugate subgroups.

Reference:

H. Grimmer, "Relations between the 14 Bravais types of lattices", submitted to *J. Appl. Cryst.*

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A series of complex hydrides based on the tetrahydroborate anion BH_4^- and crystallizing in the ABX_3 type lattice has recently been discovered. They present the first case of a family of iono-covalent hydrides that has a genuine tunable host lattice, making then an interesting new class of host materials for not only the design of hydrogen storage but also properties related to heavy metals. Amongst these, preliminary results on *REE*-based luminescence are presented in the neat and doped compounds, the Ln^{2+} -excited states surprisingly not subject to significant quenching by B-H vibrations. Unlike oxide- or halide-perovskite some members of the $\text{AB}(\text{BH}_4)_3$ group do not evolve to higher symmetries as a function of temperature. We show by means of *in-situ* synchrotron X-ray powder diffraction, vibrational spectroscopy and *ab initio* calculations in the solid state, that temperature-induced structural distortions in perovskite-type $\text{ACa}(\text{BH}_4)_3$ ($A = \text{K, Rb, Cs}$) have their origin in close di-hydrogen contacts of repulsive nature. The structural dynamics are investigated with quasi-elastic neutron scattering (QENS). These distortions are identical in symmetry to well-known lattice instabilities (soft modes) in perovskites, which generally condense to lower temperatures. Anion-substitution $\text{BH}_4^- \leftrightarrow \text{X}^-$ ($\text{X} = \text{Halide}$) calculated on ordered models can relax distortions caused by repulsive effects. High temperature phase-transitions in $\text{ACa}(\text{BH}_4)_3$ can be of first or second-order, including 2-fold superlattices, simple cubic-cubic transitions accompanied by volume expansion or complex modulated superstructures accompanied by negative volume expansion, as is the case in $\text{RbCa}(\text{BH}_4)_3$. Close di-hydrogen contacts may be suggested as a tool to tailor the crystal symmetry in complex hydride perovskites in the future.

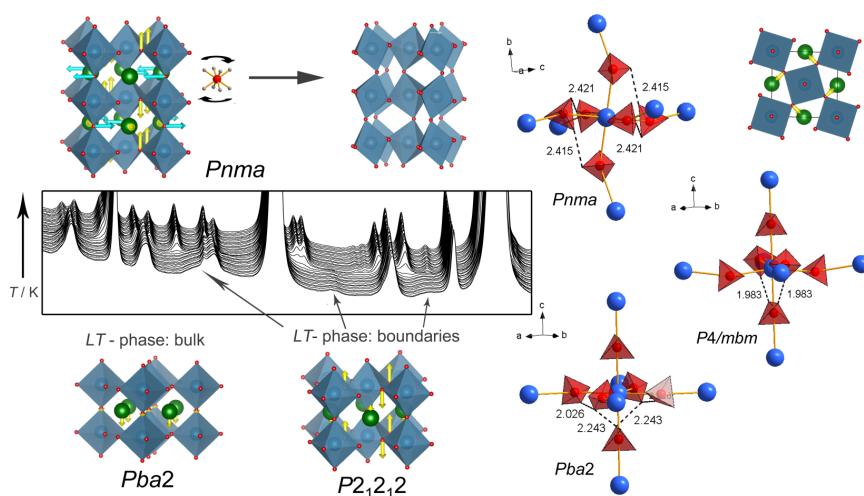


Figure 1: Distortion modes and *in-situ* diffraction data of the high-temperature phase transition in $\text{KCa}(\text{BH}_4)_3$. Important di-hydrogen contacts are marked.

P08:	Studying structure disorder in DL-Norvaline by single crystal diffuse scattering
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Diffraction methods are the most important methods to study the three-dimensional arrangement of matter at the atomic level. Real materials are often not perfectly ordered and the resulting diffraction pattern may contain a weak continuous or structured background known as diffuse scattering, in addition to sharp Bragg peaks. Our motivation is to analyse diffuse scattering in order to learn about the Short Range Order (SRO) of disordered crystals and improve the tools to model disorder phenomena.

We are now investigating the SRO in DL-Norvaline which crystallizes in three known temperature-dependent phases. At least two of them (b-phase space group C2/c above -70°C, a-phase P21/c around -90°C) show disordered average structures in which the alkyl side chain adopts several conformations [1]. The scattering data were collected using synchrotron radiation and a noise-free Pilatus pixel detector at the ESRF BM01A station. The diffraction pattern of the β -phase shows diffuse streaks parallel to a reciprocal lattice axis and diffuse clouds around low angle reflections [2] (Fig. 1). These features result from static and dynamic disorder. The diffuse streaks indicate disorder amongst stacks of layers of molecules, while the diffuse clouds arise from thermal motion. The modelling of the disorder involves the use of the Monte Carlo and differential evolution algorithms embedded in ZODS [3]. Our progress with model development will be presented.

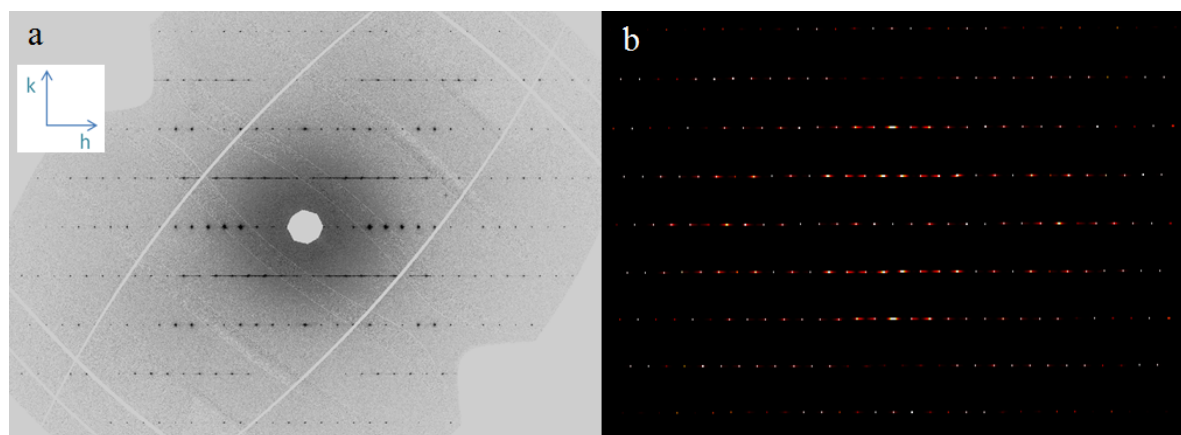


Figure 1. Diffuse scattering in the $hk0$ layer. (a) Experimental pattern (b) Simulated pattern

References:

¹ C.H. Görbitz, *J. Phys. Chem. B* **2011**, 115, 2447-2453.

² Agilent Technologies (**2012**). CrysAlisPro, Agilent Technologies, Yarnton, Oxfordshire, England.

³ ZODS: Zurich-Oak Ridge Disorder Simulation software, ETH/UZH, under development.

P09:	MEMBRANE SUPPORTS FOR X-FEL BASED PROTEIN CRYSTALLOGRAPHY
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Knowledge of molecular constituents and mechanisms underlying human diseases allows design and production of efficient medicines. Therefore, the ability to accurately and reliably determine the structure of the building blocks of life and in particular of proteins is of great importance.

The largest contribution to understanding these large molecules so far has been made by synchrotron radiation based crystallography. However, this method is limited to well-diffracting 3D crystals, which are in many cases hard to obtain and prone to damage during the exposure to X-rays. The measurements have to be carried out at low temperatures achieved using liquid nitrogen cooling systems. Moreover, this technique is not suitable for studying 2D crystals, which do not diffract in a synchrotron beam.

Free Electron Lasers (FELs) - a novel type of X-ray sources - are able to produce femtosecond (10^{-15} s) pulses of coherent radiation in a wide wavelength range, with extremely high peak brightness. They provide opportunities for a new approach to solving protein structures by serial measurements of large number of nanocrystals or 2D crystals. The short pulses allow measurements before radiation damage occurs and can be performed at room temperature.

The aim of this project is to fabricate solid supports for serial protein crystallography. These will permit a much more efficient alternative to existing solutions for delivery of the sample to the beam of the FEL. The challenge is to obtain stable and easy to fabricate membrane supports with high transparency for X-ray radiation. Therefore, suitable materials have to be selected and efficient fabrication processes established with an appropriate way of deposition of the crystals/protein solution.

References:

A. Zarrine-Afsar et al., *Acta Cryst.* **2012**, D68, 321-323.

M. Frank et al., *IUCrJ*, **2014**, 1, 95-100.

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Measurement of characteristics such as crystal structure, crystallographic orientation and grain dimensions in materials at the nanoscale is fundamental for evaluating nanocrystalline materials and correlating their structure with their mechanical properties. For such analyses, transmission electron microscope (TEM) is generally considered as the tool of choice. In TEM, the crystal structure and crystallographic orientation can be determined on a point-by-point basis. However, it is not easy to map orientations in an automated and robust manner. In 2012, Keller and Geiss demonstrated that electron backscatter diffraction (EBSD) patterns could be acquired from a thin film specimen (between 100 and 200nm) by transmitted electrons in the SEM. In such set-up, kikuchi patterns formed by these transmitted electrons come from very small volumes, which improves the lateral resolution of EBSD by more than one order of magnitude, reaching sub-10nm (Suzuki, 2013). We applied transmission EBSD (or TKD for transmission kikuchi diffraction) to characterize the microstructure of nanocrystalline electrodeposited materials. The recent advances in microtechnology require further improved mechanical properties than those of pure Ni electrodeposits, which is classically used in the fabrication of micromechanical parts through UV-LIGA process. Improvement in mechanical properties can be achieved by tuning the microstructure of materials, such as controlling the texture, grain size, nanotwins size and density. A parametric study was applied to Ni alloys and pure copper in order to link the electrodeposition parameters to the microstructure and mechanical properties of the material.

References:

R.R. Keller, R.H. Geiss, *Journal of Microscopy* **2012**, 245, 245–251.
S. Suzuki, *JOM* **2013**, 66, 1254-1263

P11: Stabilized ZrO₂: Ageing and Healing as seen by Powder X-ray Diffraction

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Due to their high ionic conductivity, stabilized ZrO₂ materials are widely used for electrolytes in solid oxide fuel cells (SOFCs) or as functional materials for oxygen sensors. Unfortunately, a degradation of the ionic conductivity of many stabilized zirconia materials has been observed under operation conditions, frequently amounting to more than 30 % over the first few hundred hours of operation. It has also been found that this degradation can be reversed to a high extent through a short high-temperature treatment of the electrolyte.

Even though the exact mechanism of the ageing process is not undisputed, better control over ageing and healing is highly desirable for industry. Accordingly, we investigated the ageing and healing properties of several compositions of Y and Sc stabilized ZrO₂ by powder x-ray diffraction, searching for a composition showing less ageing and having lower healing temperatures.

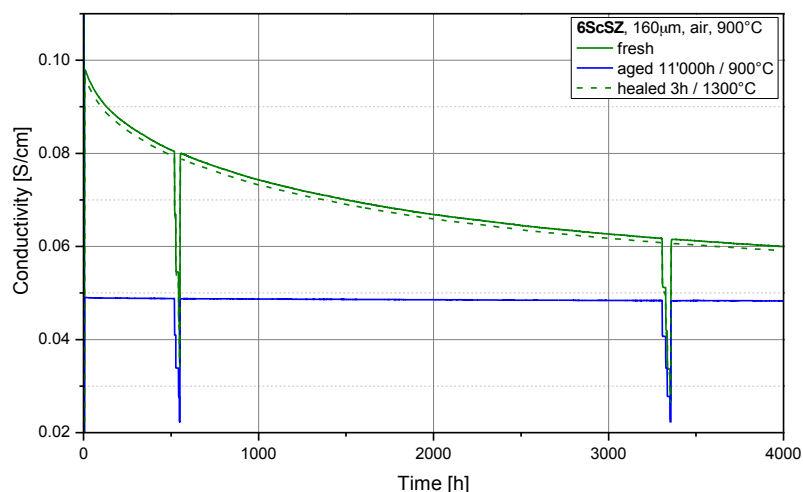


Figure 1: Conductivity degradation of a 6 %-Sc₂O₃-ZrO₂ (6ScSZ) electrolyte at 900 °C.

References:

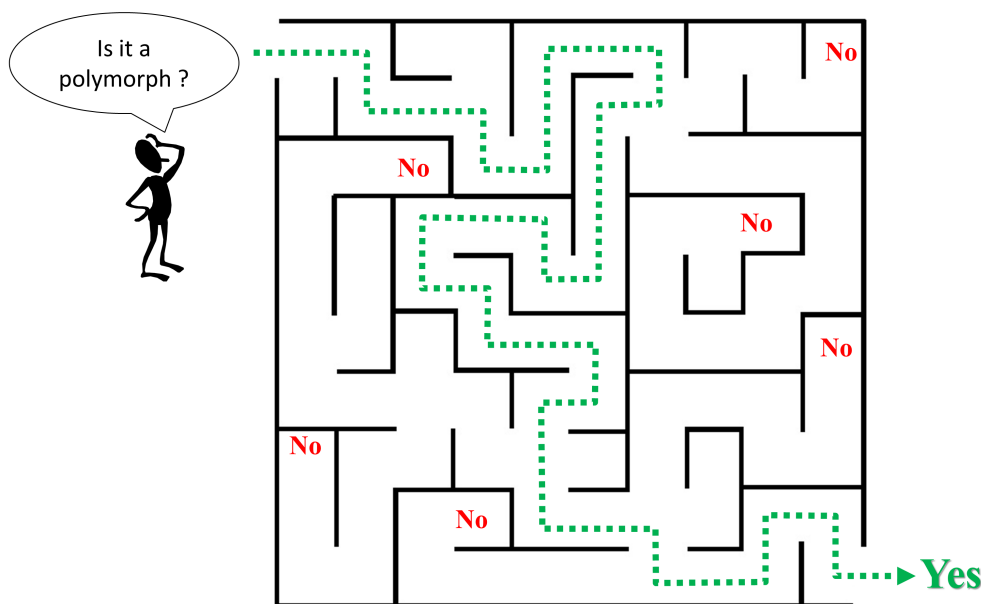
- L. J. Gauckler, D. Beckel, B. E. Buegler, et al., *Chimia*, **2004**, 58(12), 837-850.
- S. P. S. Badwal, F. T. Ciacchi, S. Rajendran, J. Drennan, *Solid State Ionics*, **1998**, 109, 167-186.
- M. R. Turner, J. A. Schuler, A. Mai, D. Penner, *Solid State Ionics*, **2014**, 263, 180-189.

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Polymorphism is a very important phenomenon not only in basic research, but certainly in pharmaceutical industry and materials science. Polymorphs possess different properties, for instance the solubility or the mechanical resistance can differ dramatically from one polymorph to the other – properties which can be crucial for their application. Hence, it is important to be able to control the formation of polymorphs and to understand their formation. We here gave some insights into the basic knowledge of polymorph formation and their identification and characterization in order to give an overview on the current state of the art. In order to give interested peoples a tool in hand to test their compounds for polymorphism, we established a series of flow sheets to follow, depending on the class of compounds, hoping that they are useful for many scientists who are not so well acquainted with polymorphism. The presented schemes resume thus the identification steps for polymorphs. It should also help to use the term polymorph correctly in order to reduce the number of publications in which this term is not used in a correct way.



Reference:

J-P. Brog, C-L. Chanez, A. Crochet, K. M. Fromm, *RSC Adv.*, **2013**, 3, 16905-16931.

P13:	XRPD study of the photoinduced HS → LS relaxation of the hydrated [Fe(pyrazine)Pt(CN)₄] polymeric network structure
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Phil Pattison,^c Andreas Hauser^a

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The 2D layer compound [Fe(pyrazine)Pt(CN)₄] has received a lot of attention because of its spin transition with a large hysteresis at room temperature [1], the possibility of bidirectional light-switching [2], and its potential as sensor for various gases [3], and in a recent study it served as test case for time-resolved electron diffraction on the nano-second time scale [4]. The present study focuses on the photo-induced HS→LS relaxation of the hydrated compound monitored by synchrotron XRPD measurements at 10 K (Figure 1). For the title compound, the time evolution of the XRD pattern shows a complex relaxation behaviour, which can be modelled with an initial random relaxation followed by a nucleation and growth process.

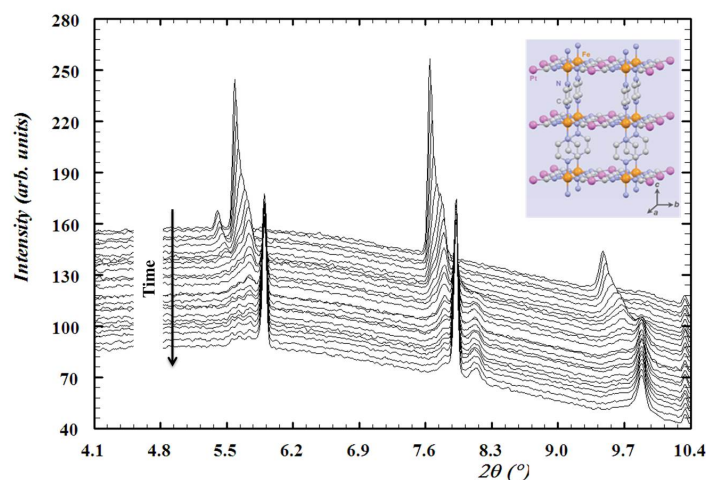


Figure 1. XRPD patterns of hydrated [Fe(pyrazine)Pt(CN)₄].2H₂O recorded at 10 K in intervals of 100s following irradiation at 532 nm for 20 minutes.

References

- [1] a) Agusti G. *et al.*, *Chemistry of Materials* **2008**, 20, 6721,
b) Cobo S. *et al.* *Angew. Chem, Int. Ed.* **2006**, 45, 5786.
- [2] Cobo S. *et al.*, *J. Amer. Chem. Soc.* **2008**, 130, 9019.
- [3] Ohba M. *et al.*, *Angew. Chem. Int. Ed.* **2009**, 48, 4767.
- [4] R. M. van der Veen, O.-H. Kwon, A. Tissot, A. Hauser, A. H. Zewail,
Nature Chemistry, **2013**, 5, 395.

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Bisisonicotinate disulphide (isoDiS, Figure 1) was widely used in our research group for Au(111) surface treatment in combination with antibacterial complexes^[1, 2]. In order to increase our knowledge on the interactions happening on the surface, crystallographic studies are performed using (mostly) the metal ions that were coated during the above-mentioned studies.

Three similar ligands were also taken into account in order to look for a better alternative and so improving the quality of the materials developed.

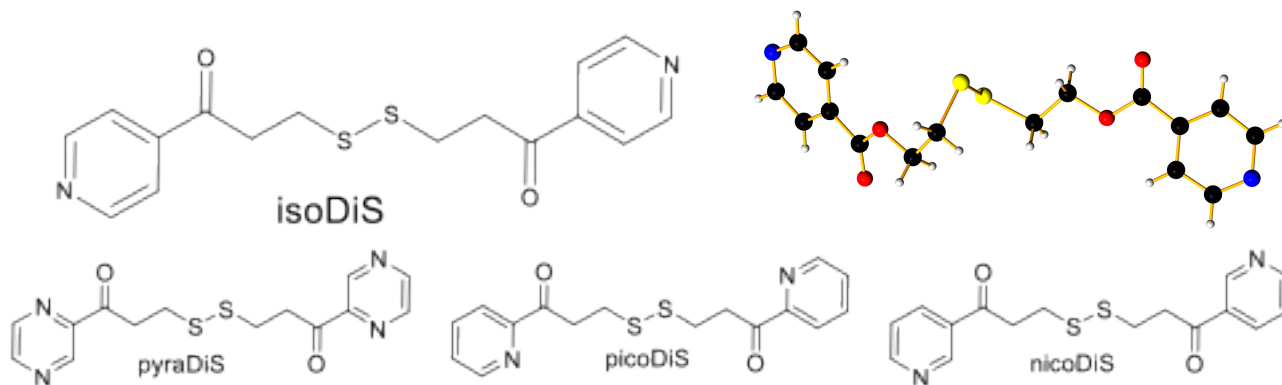


Figure 1: the 4 different ligands.

Silver(I) and copper(II) were the first two ions used (both have interesting antibacterial properties), but the study evolved quickly on other ions (e.g. mercury(II)).

Diffraction techniques are also useful to elucidate new structures of complexes that can be coated on the Au(111) surface and to prove that what we attach are really the expected complexes.

References:

- [1] M. Varisco, N. Khanna, P. S. Brunetto, K. M Fromm,
ChemMedChem **2014**, 9, 1221-1230.
- [2] P. S. Brunetto, T. V. Slenters, K. M. Fromm, *Materials* **2011**, 4, 355-367.

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Sodium ion batteries are emerging to be future energy storage devices replacing its counterpart lithium ion batteries owing to its limited geographical constraint and thereby restricting to meet the global demands. Polyanion (PO_4^{3-}) based cathode materials (NaMPO_4 , $M = \text{Mn, Fe, Co}$) for sodium ion batteries are better candidates on grounds of cycle stability, thermal stability, safety, environmental friendliness and cost.¹ The NaMnPO_4 exists in olivine and maricite phases with the former being preferable in terms of Na ion conductivity and low temperature formation while the latter is thermodynamically stable at higher temperatures.² Fig. 1 shows the structural modifications due to the different occupancy of two octahedral sites by Na^+ (green) and Mn^{2+} (red) ions. Given that there are no clear optimized explanations about the appropriate methods to synthesize NaMnPO_4 due to the complicating phase transition phenomena between maricite and olivine phases, we study NaMnPO_4 to optimize a suitable synthesis method taking into account the diffusivity of Na ions through the crystal structure.

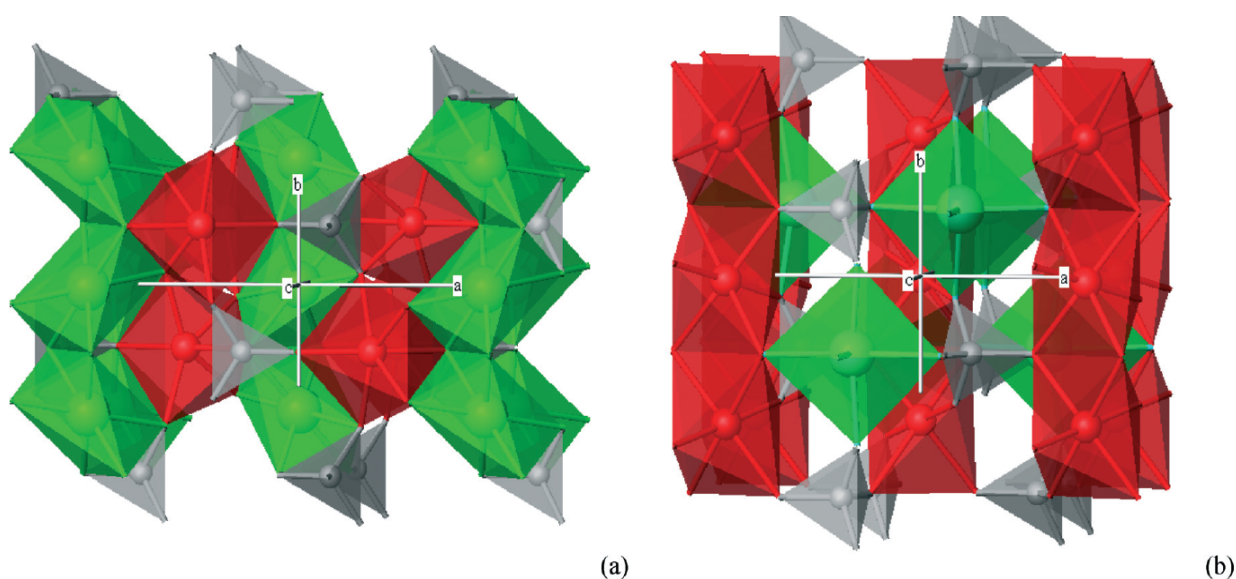


Figure 1: Schematic view of Olivine (a) and Maricite (b); MnO_6 , NaO_6 and PO_4 polyhedra are colored in red, green and grey, respectively

References:

- [1] "Recent advances in the research of polyanion - type cathode materials for Li-ion batteries", Z. Gong and Y. Yang, *Energy & Environmental Science* **2011**, 4, 3223.
- [2] "The crystal structure of NaMnPO_4 ", J. Moring and E. Kostiner, *J. of solid state chemistry* **1986**, 61, 379.

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To keep improving transistors' performances whilst reducing their gate length, the use of III-V materials is foreseen as they have much better transport properties than silicon. Their integration in future CMOS technology requires the availability of production size wafers and, therefore, the development of III-V heterogeneous integration on silicon. To that aim, high structural material quality is required. We have grown low-defective crystalline $\text{In}_x\text{Ga}_{1-x}\text{As}$ on InP substrates by Metal Organic Chemical Vapor Phase Epitaxy, using an InAlAs buffer layer to compensate for the lattice mismatch between the $\text{In}_x\text{Ga}_{1-x}\text{As}$ and InP. To enable its integration into devices, the $\text{In}_x\text{Ga}_{1-x}\text{As}$ layer was then transferred by direct wafer bonding on an $\text{Al}_2\text{O}_3/\text{Si}$ substrate¹.

The crystalline quality and the In content of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ have been frequently controlled by X-Ray Diffractometry during the process. These measurements are key for the growth as well as the wafer bonding parameters optimization and have been carried out with a D8 Discover Super Speed Solution from Bruker AXS equipped with a Cu rotating anode. A Ge (022) monochromator was mounted on the primary side and a path finder (used as analyzer or slit) was installed on the secondary side for X-Ray Diffractometry and Reciprocal Space Map measurements. For Grazing Incidence Diffractometry, Sollers slits were substituted to the path finder.

This poster presents results obtained for $\text{In}_x\text{Ga}_{1-x}\text{As}$ layers bonded on $\text{Al}_2\text{O}_3/\text{Si}$ substrates for two In contents (53% and 72% targeted) and thicknesses down to 5 nm. Applying X-Ray measurements, we have been able to verify the quality of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ transfer and in particular to check that the bonding does not impact the structural quality of the grown $\text{In}_x\text{Ga}_{1-x}\text{As}$ layers. We have also observed that for a given In content, all layers present a similar lattice parameter. To distinguish the contribution of strain and of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ stoichiometry to the lattice parameter, reciprocal space maps were performed. Up to 300 nm, the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ films are found fully relaxed: the targeted In content of 53% was achieved; measurements are still on going for the $\text{In}_{0.72}\text{Ga}_{0.28}\text{As}$ layers.

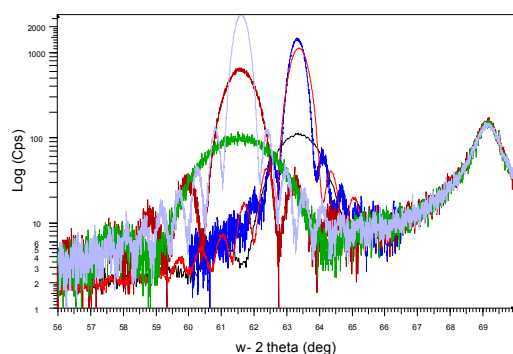


Figure 1: XRD data around the $\text{In}_x\text{Ga}_{1-x}\text{As}$ (004) peak after bonding for $x=53\%$ and 72% . The data are shown here for $\text{In}_{0.72}\text{Ga}_{0.28}\text{As}$ thicknesses of 5 nm (black), 9 nm (red) and 18.7 nm (blue) and $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ thicknesses of 6 nm (green), 13 nm (black red) and 25 nm (violet). The alignment was performed on the InGaAs peak. Out of plane lattice parameters of $6.018 \pm 0.003 \text{ \AA}$ and $5.868 \pm 0.003 \text{ \AA}$ were extracted for $\text{In}_{0.72}\text{Ga}_{0.28}\text{As}$ and $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ respectively.

Reference:

[1]: L. Czornomaz, et al., Tech. Dig.-Int. Electron Devices Meet. **2013**, pp. 2.8.1-2.8.4

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Research interest in mixed metal oxide is increasing in material science. They have multiple applications, such as batteries, ceramics, pigments, high-T_c superconductors or transparent conductors.

However, the two main challenges for the synthesis of such compounds are the lack of control on the ratio of the different metal components and the extreme conditions (up to 900 °C) that many of these oxides require during the synthesis.

To overcome these kinds of issues we propose a new strategy for the synthesis of mixed metal complexes, a “multitopic ligand approach”.

The aim is to design specific ligands with selective coordination sites to bind different metal ions. Due to the metal ion preorganization in the precursor thus formed, the stoichiometry of the final oxide material can be controlled and the extreme synthesis conditions diminished (pressure or temperature). These new mixed metal complexes will be finally combusted to oxide materials with possible new features and ideally at the nanoscale.

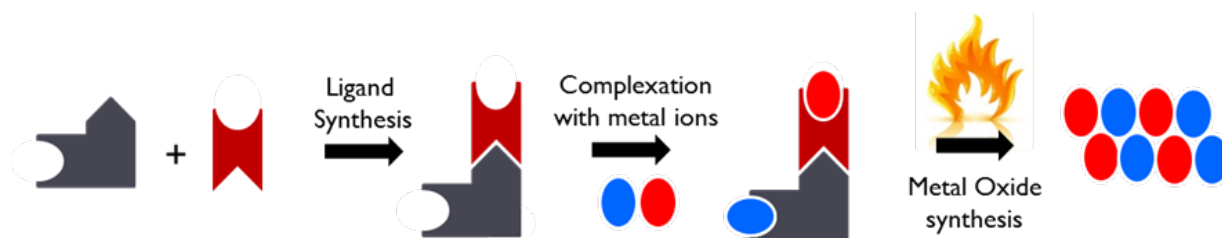


Figure 1: Multitopic ligand approach

References:

- F. Gschwind, O. Sereda, K. M. Fromm, *Inorg. Chem.* **2009**, 48, 10535-10547
 F. Gschwind, K. M. Fromm, *Z. Anorg. Allg. Chem.* **2011**, 1871-1879

P18:

ZEBRA: The new neutron single-crystal neutron diffractometer optimized for small samples and extreme conditions at SINQ

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ZEBRA, a new single crystal neutron diffractometer optimized for parametric studies and an extended q-range for crystallographic investigations, is under construction at SINQ and its commissioning is expected for 2017.

This new SINQ instrument will be capable of resolving scientific challenges emerging in systems available as small crystals only and requiring extreme sample environments. The scientific projects in focus include unconventional magnetic phases in frustrated magnets, Mott-insulating states in spin-orbit coupled systems, unconventional superconductors, functional metal-organic and perovskite materials. The ZEBRA instrument (Fig.1) will achieve high peak-to-background ratio and will host extreme sample environments. New optimized neutron delivery system (primary instrument) and new nonmagnetic high-precision sample-positioning and analyzer-detector units (secondary instrument) will be built on the position of the present TriCS single crystal diffractometer.

The project is financed by the R'Equip program of the Swiss National Science Foundation and PSI.

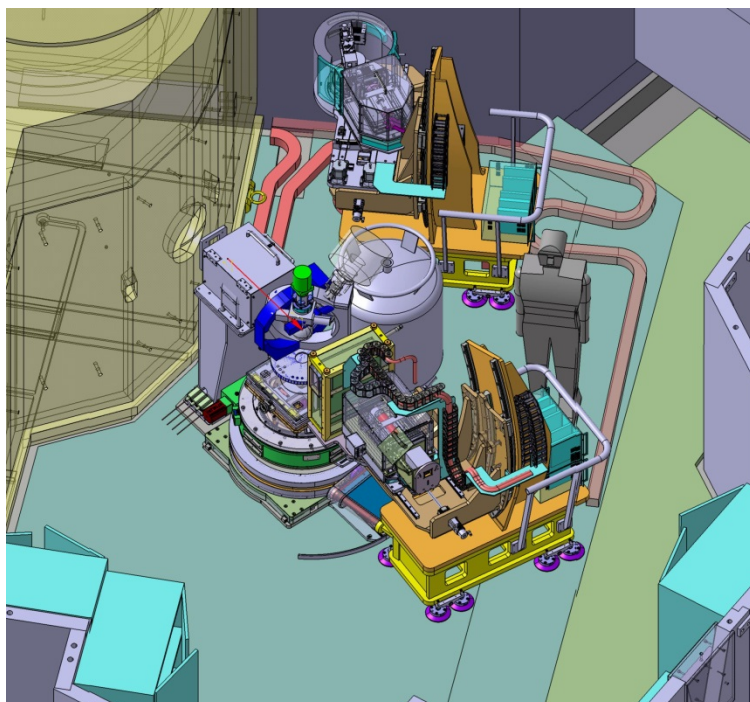


Fig. 1: Layout of the ZEBRA single crystal neutron diffractometer at the thermal beamport at SINQ. In the forward, the detector unit with 1D/2D detectors which can be exchanged fastly is shown. The second detector unit with an analyzer – allowing low background measurements – is in the parking position.

P19:	The crystallization of melt-spun poly(3-hydroxybutyrate) (P3HB) fibers investigated by WAXD
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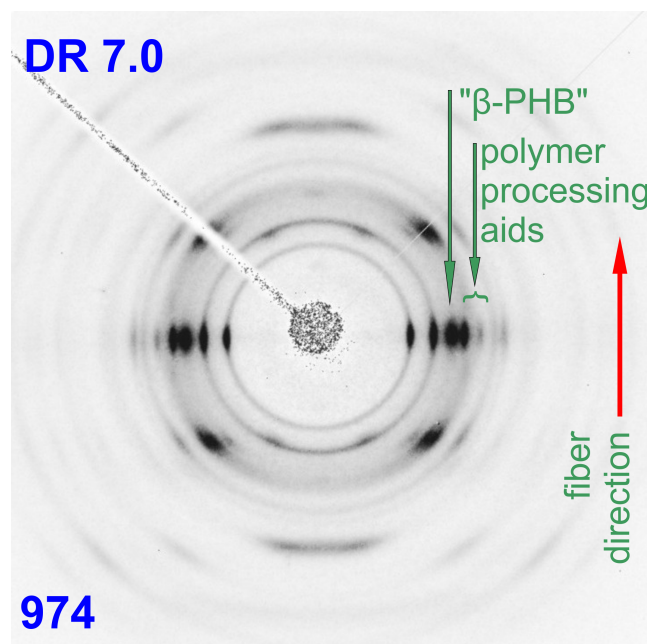
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Poly(3-hydroxybutyrate) (P3HB) is a thermoplastic polyester produced by bacteria as intracellular carbon and energy storage compound. Its relatively simple biosynthesis process leads to reasonable production costs, which makes P3HB an attractive substitute for conventional petrochemical plastics. Our goal is to develop an upscalable melt-spinning process to produce high-strength P3HB fibers, which so far couldn't be established [1]. The crystallization process is crucial in order to receive P3HB fibers with acceptable mechanical properties. Primary crystallization has to be completed during the drawing step. Secondary crystallization, that develops large spherulitic structures, has to be hindered to prevent brittleness, poor mechanical performance and conglutination of as-spun fibers.



According to the literature, P3HB can crystallize in α -form (helix) and β -form (zigzag conformation proposed). The β -form is induced by stretching, while under general conditions the α -form develops. In the WAXD pattern, the β -form is indicated by a broad additional equatorial reflection [2]. For this reflection, we found a rather particular peak shape in the equatorial 2θ scan, and we postulate for the fibers melt-spun and drawn within this work that this reflection is the trace of a highly ordered amorphous phase.

Figure 1: WAXD pattern of P3HB fiber 974 (draw ratio 7.0).

References:

- [1] R. Hufenus, F.A. Reifler, M.P. Fernández-Ronco, G. Fortunato, U. J. Hänggi (manuscript in preparation)
- [2] P. Pan and Y. Inoue, *Progress in Polymer Science* **2009**, 34(7), 605-640.

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2-(methyldeneamino)acetonitrile (MAAN) has raised some interest in the 1970ies, due to its reactivity with aminoacids and formation of peptides [1,2]. Reports on its chemical properties also included the ability to polymerize. However, results from the physical characterization of monomer and trimer appear to be scarce. In this work, we expand the solid-state characterization, and present how synergy of experimental and computational techniques can lead to a full and reliable analysis of molecular compounds. In the first step, molecular structures of MAAN and its trimer were investigated using computational techniques. In particular, conformational analyses were performed using a stochastic search. This resulted in optimized molecular structures of both monomer and trimer. The second step involved characterization of commercial sample. Infrared and Raman spectra were measured, but straightforward monomer-trimer disambiguation was not possible. Therefore, for both monomer and trimer, (an)harmonic vibrational frequencies were calculated using the density functional theory and coupled cluster theory. Based on the comparison between the experimental and calculated vibrational spectra, it was found that the sample contains MAAN in trimeric form. This information was used in the third step of this study - structure determination from XPD data. Namely, although the measured pattern was of excellent quality, reciprocal structure determination methods did not work, so the knowledge of molecular entity and its optimized geometry was crucial. Trimer molecules was optimized in direct-space, using the program FOX [3]. Restrained Rietveld refinement of the resulting structure was carried out using the XRS suite [4]. As last, computation techniques were used again, to minimize the energy of the crystal cell, and thus serve as a validation tool of the experimentally obtained crystal structure.

References:

- [1] A.S. Subbaraman, Z.A. *et al.* *Origins of Life* **1975**, 6, 537–539.
- [2] K. Kawashiro, K. Nishiguchi, Nara, T. *Orig. Life Evol. Biosph.* **1989**, 19, 133–142.
- [3] Favre-Nicolin, V., Černý, R. *J. Appl. Cryst.* **2002**, 35(6), 734–743.
- [4] Baerlocher, Ch, Hepp, A. XRS suite, ETH Zurich

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Several examples of challenging applications were selected to highlight the performance of PHOTON100 at customer sites and in the Bruker application laboratories.

CMOS technology based X-ray detectors offer numerous advantages compared to traditionally used CCD detectors:

- CMOS sensors are available in larger sizes
- CMOS sensors provide excellent signal-to-noise ratios even when only moderately cooled. This allows the design of air-cooled detectors. Combined with low power consumption, this leads to minimized pre-installation requirements and extended life time
- while CCDs use a bucket-brigade principle for reading-out, CMOS technology does allow for direct and therefore continuous sensor read-out

The capability to continually read out pixels provides a new approach for data collection. While CCDs require closing the shutter for each read-out step, introducing system overhead, CMOS based detectors can be operated in shutterless mode, which not only eliminates over-head time but it also reduces mechanical jitter.

We will present details on examples from poorly diffracting MOF crystals, an incommensurately modulated phase of Cr₂P₂O₇, as well as multipolar refinement, using high resolution data beyond 0.45 Å

Calls for proposals

Beside normal proposals, most facilities allow urgent beam time requests. Please check directly with the facility.

Facility	Deadline(s)	Link
SLS: Swiss Light Source All except PX lines Protein crystallography beamlines (PX)	March 15, Sept. 15 Feb. 15, June 15, Oct. 15	www.psi.ch/useroffice
SINQ: Swiss Spallation Neutron Source All instruments (regular calls)	May 15, Nov. 15	www.psi.ch/useroffice
SINQ/SLS Joint x+n proposals (MS/HRPT)	Feb. 15, 2015	www.psi.ch/useroffice
SpS: Swiss Muon Source All instruments	Dec. 9, June 10	www.psi.ch/useroffice
ESRF: European Synchrotron long term proposals short term proposals	Jan. 15 March 1, Sept. 1	www.esrf.eu/ UsersAndScience/
ILL: Institut Laue Langevin All instruments	Feb., Sept.	www.ill.eu
FRM II: Heinz Maier-Leibnitz All instruments	May 02, 2014	user.frm2.tum.de
SNS Spallation Neutron Source Oak Ridge	various	neutrons.ornl.gov

Calendar of forthcoming meetings

(Please mail the missing information on meetings of interest to Jurg.Schefer@psi.ch)

				Abstract Deadline
2014				
Aug. 28-Sep. 06	Pavia Italy	1st European Crystallography School 2014 http://2014.aicschool.org		16.06.2014
Sep. 1-5	Roskilde Denmark	35th Risø International Symposium on Materials Science http://www.dgm.de/dgm/mse-congress/		06.01.2014
Sep. 8	Dübendorf CH	2014 Annual Meeting of the SGK / SSCr		31.07.2014
Sep. 21-24	Jena Germany	92nd Annual Meeting: Deutsche Mineralogische Gesellschaft http://www.dmg2014.de		30.05.2014
Sep. 21-26	Ile d'Oléron France	JDN22 - School "Crystallography And Neutrons" http://www-llb.cea.fr/JDN22/		30.06.2014
Sep. 23-25	Darmstadt Germany	Materials Science and Engineering (MSE 2014) http://www.dgm.de/dgm/mse-congress/		17.02.2014
Oct. 5-8	Ellwangen Germany	Summer school " <i>Theory and Practice of Modern Powder Diffraction</i> " http://www.kofo.mpg.de/iycr/index.html		31.03.2014
Oct. 5-14	Grindelwald CH	10th World Conference on Neutron Radiography (WCNR-10) http://indico.psi.ch/conferenceDisplay.py?confId=2019		17.03.2014
2015				
Jun. 7-20	Zurich Switzerland	Zurich School of Crystallography – Bring Your Own Crystals http://www.chem.uzh.ch/linden/zsc/		16.01.2015
Jun. 14-18	Covalese- Trentino, Italy	DSE 2015 – 100 Years of Debye Scattering Equation http://dse2015.wix.com/dse2015		to be announced
Jun. 30 –Jul. 03	Lucerne Switzerland	5th European Pefc & H2 Forum http://www.efcf.com		to be announced
Jul. 1-4	Lucerne Switzerland	11th European SOFC & SOE Forum http://www.efcf.com		to be announced
Aug. 23-28	Rovinj Croatia	ECM-29 2015 http://www.ecm29.org		to be announced
2016				
28. Aug-01. Sep	Basel CH	European Crystallographic Association, ECM-30 http://www.ecm30.org		to be announced
2017				
28. Aug-01. Sep	Hyderabad India	IUCr-2017, 24 th General Assembly and Congress of IUCr http://www.iucr2017.org		to be announced

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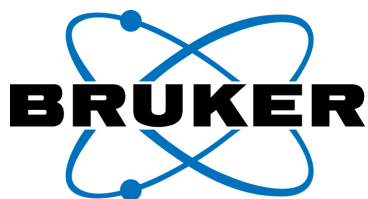
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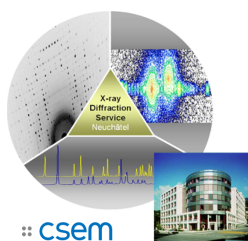
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