Danscatt Annual meeting 2017
1st – 2nd of June 2017

At Department of Physics, Chemistry and Pharmacy,
SDU, Odense

SDU
Organizers:
Main organizer: Associate Professor Dorthe B. Ravnsbæk
Co-organizer: Associate Professor Beate Klösgen

Program committee:
Dorthe B. Ravnsbæk, SDU
Beate Klösgen, SDU
Michael Gajhede, CU
Dorthe Posselt, RUC
Martin M. Nielsen, DTU

PhD award evaluation committee:
Dorthe B. Ravnsbæk, SDU
Søren Skou Thirup, AU
Erik Lauridsen, Xnove Technology APS

Sponsors:
Xnovo Technology ApS
JJ X-ray
LINX
Ramcon
# Content

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Program 1\textsuperscript{st} of June</td>
<td>3</td>
</tr>
<tr>
<td>Program 2\textsuperscript{nd} of June</td>
<td>5</td>
</tr>
<tr>
<td>Practical information about the venue</td>
<td>6</td>
</tr>
<tr>
<td>Venue map</td>
<td>7</td>
</tr>
<tr>
<td>Talk abstracts</td>
<td>9</td>
</tr>
<tr>
<td>List of posters</td>
<td>25</td>
</tr>
<tr>
<td>List of participants</td>
<td>29</td>
</tr>
<tr>
<td>Poster Abstracts</td>
<td>33</td>
</tr>
</tbody>
</table>

We are very happy to have you at SDU
<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
<th>Presenter</th>
<th>Topic</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:30-12:00</td>
<td>Registration and sandwiches</td>
<td>Old Friday Bar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12:10</td>
<td>Meeting opening</td>
<td>U110</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12:20</td>
<td>Session 1: XFEL</td>
<td>U110</td>
<td>Invited speaker: Martin M. Nielsen, DTU</td>
<td>XFEL experiments on metal complexes</td>
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<td>Invited speaker: Dorthe B. Ravnsbæk, SDU</td>
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<td>Session 1: XFEL</td>
<td>U110</td>
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<td>Imaging Macromolecules with X-ray Laser pulses</td>
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<td>Coffee break</td>
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<td>13:50</td>
<td>Session 2</td>
<td>U110</td>
<td>Anine Borger, KU</td>
<td>Will wide angle X-ray scattering (WAXS) reveal the molecular alignment of polystyrene stretched in its melted state</td>
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<td>14:05</td>
<td>Chairman: Dorthe Posselt, RUC</td>
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<td>14:20</td>
<td>Invited speaker: Henriette W. Hansen, RUC</td>
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<td>Dynamics in glass-forming liquids under pressure from second to picosecond</td>
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<tr>
<td>14:35</td>
<td>Invited speaker: Thomas Christiansen, KU</td>
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<td>Studying ancient Egyptian black ink under the synchrotron lights</td>
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<td>14:50</td>
<td>Invited speaker: Kim K. Rasmussen, KU</td>
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<tr>
<td>14:50</td>
<td>PhD award ceremony</td>
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<td>15:00</td>
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<td>Session 3</td>
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<td><strong>Chairman:</strong> Martin Bremholm, AU</td>
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<td><strong>Christine J. McKenzie, SDU</strong></td>
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<td>_Halogen Bonding Traps an Iron Catalyst-</td>
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<td><em>Hypervalent Iodine Adduct</em></td>
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<td><strong>Steinar Birgisson, AU</strong></td>
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<td>_Unraveling the reaction mechanism during</td>
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<td><em>hydrothermal formation of α- and β-MnO₂</em></td>
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<td><strong>Kasper S. Pedersen, DTU</strong></td>
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<td>_Elucidating the Electronic Structure of</td>
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<td><strong>Invited speaker:</strong> Kim Lefmann, KU</td>
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<td>_Quantum Materials studied by inelastic</td>
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<td><em>neutron scattering</em></td>
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<td>17:00</td>
<td>Poster session</td>
<td>Old Friday Bar</td>
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<td>19:00</td>
<td>Dinner</td>
<td>SDU Restaurant</td>
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## Program 2/6-2017

| Session 4 | Room: 110 | Invited speaker: Robert Feidenhans'l, XFEL  
*The European XFEL* | 9:00 |
|---|---|---|---|
| Chairman: | Beate Klösgen, SDU | Invited speaker: Savvas Savvides, Ghent University  
*Mammalian crystalline proteins in vivo see the light* | 9:30 |
| Coffee break | Room: 110 | Chairman: | Kell Mortensen, KU |
| Location: Winter Garden | Invited speaker: Henning Friis Poulsen, DTU  
*Danscatt status* | Invited speaker: Fredrik Melander, Ministry of Higher Education and Science  
*Radiation report and ESS strategy* | 10:20 |
| 9:00 | 9:30 | 10:00 | 10:20 |
| Coffee break | Room: 110 | Chairman: | Kenny Ståhl, DTU |
| Location: Winter Garden | Invited speaker: Mads Ry, DANMAX  
*DanMAX – The Danish Materials Science Beamline at MAX IV* | Invited speaker: Hanna Wacklin, ESS  
*Soft Matter Science at ESS* | 11:05 |
| 11:05 | 11:30 | 12:00 | 11:20 |
| Sandwiches | Room: 110 | Chairman: | | 12:30 |
| Location: Old Friday Bar | Invited speaker: | Invited speaker: | | |
Practical information about the venue

All the locations for the Danscatt Meeting are located along the central (and almost 1 km long) hallway at SDU called “Gydehutten” – see map on the next page.

Registration, lunch and the poster session will be in “the Old Friday Bar”. The nearest entrances are B, F, K, N and O. Note that if you use entrance B, F and K you need to take the stairs up one level after entering.

The scientific sessions will be in lecture hall U110, which is located close to Entrance A. Lecture hall U110 is at street level. Thus, if you are on “Gydehutten”, e.g. when going from lunch to the sessions, you need to take the stairs down one level.

The coffee breaks will be in “the Winter Garden”, which is located on “Gydehutten” right across the stairs leading down to lecture hall U110.

The dinner on the 1st of June will take place in “the SDU Restaurant” by the main entrance. Note that the dinner will start at 19:00. After the dinner (starting from 20:30) it will be possible to hang out (and buy beer) in the restaurant. The restaurant closes at 23:00.

Wifi:
Besides Eduroam, SDU offers an open network named “SDU_Guest”. No login required for the SDU_Guest network.

Getting to and from SDU:
Bus lines 41-44 connect SDU and downtown Odense (stops at the railway station). The ticket costs 24,- dkr. You can buy a ticket on the bus (they only accept coins – preferable precise amount) or by using your mobile through https://www.fynbus.dk/sms-billet. The trip takes approximately 20 minutes. Upon arrival at SDU the bus stops at the Main Entrance and Entrance C. When departing from SDU the bus leaves from Entrance B and the main Entrance. Note that in the evening the two last buses leave at 22:20 and 23:34.

Alternately, you can take a taxi to and from SDU (Campusvej 55). The phone number for Odense Taxi is +45 6615 4415.

Parking at SDU:
When parking at SDU you need to make a digital parking registration via the self-service Ipads placed at the building. Once you have entered your registration number your permit will be valid until 23h59 on the same day.
Talk abstracts
XFEL experiments on metal complexes
Martin Meedom Nielsen*
Department of Physics, Technical University of Denmark, 2800 Kgs. Lyngby

*Corresponding author: mmee@fysik.dtu.dk

XFELs are revolutionizing the experimental capabilities in physical chemistry, biology, and condensed matter physics. At the same time, they also pose new challenges for the experimenters. Unlike synchrotrons, each X-ray pulse is unique in the sense that wavelength, wavelength distribution and intensity may vary from shot to shot. Hence, we need to record and characterize the response from each individual X-ray bunch, which has implications for experiment design, detectors, data handling, and data rates, which may reach several 10s of TB per experiment. Here I will show some examples of our experience from 7 years of hard X-ray XFEL experiments, including a ‘behind the scenes’ look at what it is like to do such experiments, and some of the results, we obtained. A common theme in our work has been tracking ultrafast energy and charge transfer in solvated metal complexes and the interaction between solute and solvent. An example is shown in the figure below [nat. comm. 2016, DOI: 10.1038/ncomms13678]:

The efficiency, selectivity and rate of chemical reactions depend critically on the reaction environment. Solvation, the local organization of the solvent molecules around a solute, has a central role in the description of condensed phase chemical properties. Through XFEL experiments, we could track the sub picosecond light-induced motion of a photocatalytically active molecule and the structural response of the solvent to the changes in the molecular structure, shedding light on the atomistic mechanistic processes of solvation,
Imaging Macromolecules with X-ray Laser pulses  
Henry N. Chapman*

Center for Free-Electron Laser Science, DESY, Notkestrasse 85, 22607 Hamburg, Germany  
Department of Physics, University of Hamburg, Luruper Chaussee 149, 22761 Hamburg, Germany  
Centre for Ultrafast Imaging, Luruper Chaussee 149, 22761 Hamburg, Germany  
*Corresponding author: henry.chapman@desy.de

The pulses from X-ray free-electron lasers are a billion times brighter than the brightest synchrotron beams available today. When focused to micron dimensions, such a pulse destroys any material, but the pulse terminates before significant atomic motion can take place. This mode of “diffraction before destruction” yields high-resolution structural information from proteins that cannot be grown into large enough crystals or are too radiation sensitive for high-resolution crystallography. This has opened up a new methodology of serial femtosecond crystallography for radiation damage-free structures, possibly time-resolved, without the need for cryogenic cooling of the sample.

Crystallinity still comes with the drawbacks of reduction in structural information due to the insufficient Bragg sampling of diffraction data. Single particles, in contrast, give rise to continuous diffraction patterns that may be readily phased to directly form an image. There is thus a high motivation for single molecule diffraction, but even with the power of the X-ray FEL, diffraction signals are low.

One way to address the low diffracted signals of single molecules is to place many oriented particles into the beam so that their diffraction signals sum to give a measurable pattern, but without translational symmetry that gives rise to Bragg peaks. We have shown this can be achieved by using crystals with translational disorder. We have found that crystals of large macromolecules may possess such translational disorder. This has previously limited the achievable crystallographic resolution in many cases, but we have managed to measure the much weaker continuous diffraction from crystals of photosystem II, shown in Fig. 1, which extends far beyond visible Bragg peaks. We have reconstructed an image of this macromolecular complex using the phasing approach of single-molecule diffraction.

Diffraction measured from disordered photosystem II crystals at LCLS (left) and the reconstructed density obtained by iterative phasing (right). From Ayyer et al. Nature 530, 202 (2016).
Investigating membrane protein structure using match-out deuterated detergents and SANS

Structural knowledge of membrane proteins is paramount to the understanding of a wide range of questions in life science. In recent years, a number of alternatives to traditional crystallization have emerged, among them the use of various lipid:protein particles in connection with small-angle scattering and electron microscopy, permitting studies of membrane proteins in solution. The molecules or particles used to screen the hydrophobic membrane spanning region from the solvent are, however, highly visible in the data and complicate the data analysis significantly.

Here, a new approach is presented using match-out deuterated analogues of commonly used membrane protein storage detergents in combination with SANS, allowing for structural studies of membrane proteins without the nuisance signal from detergents. Recent results on a wide variety of different membrane proteins are presented. This method does not rely on the expensive and time-consuming approach of producing perdeuterated membrane proteins or deuterated lipids in vivo. Furthermore, as many membrane protein structures based on crystallography are of medium resolution at best, the resolution obtainable by using SANS can be very useful. In the future, this technique will be very applicable at facilities like ESS as the high flux and time-of-flight can be utilized to obtain good data on small unstable samples.

Figure 1: SANS data from Photosystem I (PSI, green), and from the ionotropic glutamate receptor A2 (GluA2, yellow). The signals come solely from the proteins, since match-out deuterated detergents were used.
Integrative structural biology of the genetic switch of TP901-1


1Biological Chemistry, Department of Chemistry, University of Copenhagen
2University Alpes, CNRS, CEA, Institut de Biologie Structurale, Grenoble, France
3Metabolic signalling and regulation, Department of Bioengineering, Technical University of Denmark, DK-2800 Lyngby, Denmark

*Corresponding author: Leila@chem.ku.dk

Virus particles that infect bacteria - also known as bacteriophages - have gained attention with the still increasing threat from multi-resistant bacteria, such as Pseudomonas. Bacteriophages can be classified into two categories: lytic and temperate bacteriophages. For phage therapy, lytic bacteriophages are most desirable, whereas temperate bacteriophages offer other application possibilities such as genetic toggle switches (Gardner, T.S. et al. 2000).

Bacteriophage TP901-1 is an example of a temperate bacteriophage, and infection of Lactococcus lactis can result in two outcomes for the infected cell. Either the cell will eventually lyse (lytic life cycle) and release new phage progeny to its surroundings, or the cell survives as lysogens by incorporation of the TP901-1 genome into that of the bacterium. The TP901-1 lysis-lysogeny decision is controlled by a genetic switch consisting of two promoters (PL, lytic and PR, lysogenic), three operator sites OR (right), OL (left) and OD (distant), and two genes cl (Clear I) and mor (Modulator Of Repression) coding for two proteins. The hexameric repressor CI (6x180 aa), binds as a dimer of trimers to OR, OL and OD, and is responsible for the lysogenic life cycle (Fig. 1a), whereas the anti-repressor MOR (74 aa) interacts in a so far unknown way with CI in order to allow transcription from PL, the lytic cycle (Pedersen, M. et al. 2008) (Fig. 1b).

Previously we have determined the crystal structure of the DNA binding domain CI-NTD in complex with the OL half site (Frandsen, K.H. et al. 2013). Also we have described the dynamics of a truncated variant of CI, CIΔ58, and modelled the dimer from SAXS data (Rasmussen, K.K. et al. 2016). We have now by crystallography solved part of CTD, CTD1, and can describe the whole CIΔ58 with high-resolution structures. This enables us to model the CIΔ58 interaction with DNA from SAXS data. We also attempted to obtained X-ray structure of the modulator, MOR, but unsuccessfully so far. Instead we have employed NMR to solve the structure of MOR and map the CIΔ58:MOR binding sites. Furthermore, based on native mass spectrometry and NMR, we can show that the CIΔ58:MOR and CIΔ58:OL complexes are mutually exclusive, supporting the hypothesis of a competition between CI and MOR as determinant of the cell fate.
Will Wide Angle X-ray Scattering (WAXS) reveal the molecular alignment of polystyrene stretched in its melted state?

Anine Borger\textsuperscript{a*}, Kell Mortensen\textsuperscript{a}, Jacob Kirkensgaard\textsuperscript{a}, Ole Hassager\textsuperscript{b}, Qian Huang\textsuperscript{b}, Kristoffer Almdal\textsuperscript{c}

\textsuperscript{a) Niels Bohr Institute, University of Copenhagen}
\textsuperscript{b) Dept. Chemical Engineering, Danish Polymer Center, Technical University of Denmark}
\textsuperscript{c) Dept. of Micro- and Nanotechnology, Technical University of Denmark}

*Corresponding author: aborger@nbi.ku.dk

From a materials science point of view it is highly important to understand the link between the response to macroscopic deformation of current polymer materials and the molecular structure on the length scale of the polymer coils as well as that of the monomers and their interactions. Such information will enable us to design future polymer materials with novel desired properties.

Current commercial polymer materials are polydisperse, which means that the distribution of chain lengths is relative broad. This study investigates the effects of such dispersity by probing how the presence of longer chains affects the response to deformation of a short-chain model system, i.e. a bi-disperse melt of well-defined long and short chains compared to a monodisperse model system of short chains. The experiment is carried out in two steps: The first step is to stretch the sample and allow it to relax while measuring its response using a Filament Stretch Rheometer, which both makes the deformation and measure the rheological parameters. After the response measurement, the sample is quenched below its glass-transition temperature, with the aim to preserve the molecular conformation for the further studies. The second step is to measure the molecular alignment of the polymer molecules in the quenched samples using Small-Angle Neutron Scattering for the overall coil conformation, and Wide-Angle X-ray Scattering to probe the structure on the length scale of the monomers.

The presence of the long chains enhances the order in the system and preserves it through longer relaxation time. This observation is quantified, and ideas for future work are discussed.
Dynamics in glass-forming liquids under pressure from second to picosecond
Henriette Wase Hansen\textsuperscript{1*}, Alejandro Sanz\textsuperscript{1}, Karolina Adrjanowicz\textsuperscript{2}, Bernhard Frick\textsuperscript{3} and Kristine Niss\textsuperscript{1}
\textsuperscript{1}Glass & Time, IMFUFA, Department of Science and Environment, Roskilde University, Denmark, \textsuperscript{2}Institute of Physics, University of Silesia, Katowize, Poland, \textsuperscript{3}Institut Laue-Langevin, Grenoble, France
\*Corresponding author: hwase@ruc.dk

We present a combined high-pressure cell for doing simultaneous dielectric spectroscopy and neutron scattering in the range 1-300 K, 0.1-500 MPa. We study both the fast and slow dynamics simultaneously in glass-forming liquids and are able to see signal with both techniques. We follow the fast and slow dynamics along isobars, isotherms, and in particular along isochrones, i.e. constant relaxation time $\tau_\alpha (T, P)$.

We use the cell to test the isomorph theory. The fundamental prediction of the isomorph theory is the existence of isomorphs, which are curves in the phase diagram along which all dynamical phenomena and structure are invariant [1]. The alpha relaxation, both its time and spectral shape, is one of the properties that is invariant along an isomorph, and we can therefore experimentally identify the isomorphs by the isochrones. We therefore expect to see isochronal superposition along the isochrones, i.e. all types of spectra should collapse.

We explore the dynamics on a range of different timescales from picosecond to second timescales, and show the difference between a van der Waals liquid and a H-bonding liquid along the glass transition isochrone, i.e. $\tau_\alpha = 100$ s, and higher temperature isochrones, all found from dielectric spectroscopy.

References
Studying ancient Egyptian black ink under the synchrotron lights

Dr. Thomas Christiansen
Department of Cross Cultural and Regional Studies, Egyptology Section, UCPH
thochr@hum.ku.dk

In the spring 2016, a research team from the University of Copenhagen, including chemists, physicists and Egyptologists, was granted beam-time at the X-ray microscopy beam-line ID21, the European Synchrotron Radiation Facility (ESRF), in order study the chemical composition of ancient Egyptian inks and papyri from the Papyrus Carlsberg Collection (UCPH). Carefully selected papyrus fragments from the collection were analyzed using µX-ray fluorescence (XRF) and µX-ray absorption near edge structure spectroscopy (XANES). We encountered hitherto unknown types of ink containing metalloids in a number of fragments coming from ancient Egypt, amongst others a copper containing carbon ink as seen in the figure.

Greek papyrus from Egypt dated to the 17th of September. The green frame insert marks the area that was scanned with a focused beam (160x100 steps of 5µm) and the black circles the regions from where XANES data was obtained: the ink, the fibers and single particles. The XANES spectra to the right show that the Cu in the ink is characterized by cuprite, malachite and azurite.

These inks are the earliest examples in the history of writing of the conscious addition of metal compounds during the manufacture of black ink. From our results it seems that the raw material for some of the “refined” black inks in the ancient Mediterranean were obtained as byproducts of metallurgy, glaze and glass production.

The results obtained so far using the synchrotron radiation from ID21 in the study of the black ink on ancient papyrus manuscripts provide exiting new perspectives on a decisive chapter in the history of science: the invention and evolution of ink. Further, the chemical and structural signatures obtained through the X-ray analysis reflect the physical properties of the manuscripts and thereby address one of the central challenges facing the historian: the fact that the majority of ancient manuscripts lack a recorded archaeological context. It is our expectation that the “finger-prints”, we have found, in the future will contribute to the mapping of characteristic traits of ink and papyrus, which is of importance for their chronological and geographical origin.
Hypervalent iodine compounds like iodosylbenzene (PhIO) and iodylbenzene (PhIO₂) are important reagents for carrying out selective oxygen atom transfer reactions. Even though oxidations by PhIO are typically catalyzed by metal catalysts, until our report of an iron complex of PhIO¹,² no metal catalyst-PhIO adduct had previously been structurally characterized. The trick for capturing this highly reactive compound proved to be its stabilization by multiple and strong halogen-bonding interactions, Figure (a). Intradimer I…O distances are up to 1 Å shorter than the sum of the van der Waal radii of O and I, and the shortest of them is by a relatively large margin the shortest non-covalent O…I interaction in the CSD.

(a) Single crystal X-ray structure shows dimers of a dicationic non-heme iron complex with monomeric PhIO coordinated to the seven coordinated iron atom. (b) XANES shows I oxidation state in the Fe-OIPh complex is reduced compared to native (PhIO)n.

For over a century PhIO has been reported as being amorphous, thus a comparison of this unique iron complex with parent uncoordinated PhIO was hampered by the fact the crystal structure of PhIO was not known. We have now determined the crystal structure of polymeric PhIO along with a new polymorph of PhIO2 using PXRD,³ and using iodine L₃-edge XANES Figure (b)³ show that the iodine oxidation state is reduced in the catalyst-PhIO adduct compared to (PhIO)n.

Supramolecular halogen bonds are well established for the solid and solution states. Our studies of the Fe-OIPh complex and hypervalent iodine compounds have revealed that the halogen bonding interaction is strong enough to survive high vacuum gas phase conditions.²,⁴

Unraveling the reaction mechanism during hydrothermal formation of α- and β-MnO₂

Steinar Birgisson,* Dipankar Saha and Bo Brummerstedt Iversen.

Center for Materials Crystallography, Department of Chemistry and iNANO, Aarhus, Denmark *Corresponding author: steinar@chem.au.dk

MnO₂ is a multifunctional material finding applications in catalysis, molecular sieving and electrochemistry.¹⁻⁴ Materials properties of MnO₂, such as crystal structure, microstructure and exact chemical composition play a vital role in determining its performance in different applications. It is therefore of high interest to be able to control those material properties through the material synthesis. α-, β- and δ-MnO₂ nanocrystals can be synthesized hydrothermally in a redox reaction between KMnO₄ and MnSO₄.⁵ Here the synthesis is investigated using in situ powder X-ray diffraction (PXRD) and in situ total scattering/pair distribution function (TS/PDF) analysis. Sequential Rietveld refinements of the in situ PXRD data show that α-MnO₂ nanoneedles are formed initially. Upon prolonged reaction time the α-MnO₂ nanoneedles undergo a solid state phase transformation to β-MnO₂ nanoneedles. The size and aspect ratio (needle length over needle diameter) of both α- and β-MnO₂ can be controlled to some extent by varying reaction time and temperature. Sequential real space Rietveld refinements of the in situ PDF data show that the precursor solution, i.e. the mixture of KMnO₄ and MnSO₄ before heating, consists of disordered δ-MnO₂ nanocrystals and an amorphous MnO₂ phase exhibiting a local structure similar to α-MnO₂ (α-MnO₂(A)). Upon heating the δ-MnO₂ nanocrystallites transform to α-MnO₂ in a solid state mechanism. Thereafter the α-MnO₂(A) is consumed in growing the α-MnO₂ nanoneedles until all of them have been transformed to β-MnO₂. β-MnO₂ also grows by inclusion of α-MnO₂(A) during and shortly after the phase transformation. Afterwards, all consumption of α-MnO₂(A) stops and further β-MnO₂ growth progresses via Ostwald ripening. Interestingly, the weight fraction of α-MnO₂(A) is still ~45% at that point (after 25 minutes of reaction at 250°C) showing that phase pure product is never obtained. Combination of the information collected from PXRD and TS/PDF analysis allows for a proposal of a possible reaction mechanism as summarized in the figure.

Elucidating the Electronic Structure of Heavy Transition Element Ions by Hard X-ray Magnetic Circular Dichroism Spectroscopy

Kasper S. Pedersen1,*, Andrei Rogalev2, Anna Efimenko,2 Fabrice Wilhelm2, Katharina Ollefs2, Alain Tressaud3, Etienne Durand3, Rodolphe Clérac4
1 Department of Chemistry, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark
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The interest in the heavier, 4d and 5d, transition metal ions recently blossomed due to the observation of exotic quantum phenomena in their oxides.1,2 Their singular properties originate at the microscopic level from the presence of exceedingly strong spin-orbit coupling within the 5d electronic configuration. Modeling of the magnetic behavior of such systems is exceedingly challenging and requires state-of-the-art experimental techniques that can probe locally the electronic states, the spin and the orbital atomic magnetic moments. These could be directly determined from Resonant Inelastic X-ray Scattering (RIXS) and X-ray Magnetic Circular Dichroism (XMCD) measurements at the L2,3 absorption edges of the 5d elements. Herein, we present recent measurements on a series of molecular 5d metal ion complexes. Our experimental data taken at magnetic field up to 17 Tesla and at temperatures from 2 K to 300 K allowed us to obtain unprecedented insight into the electronic and magnetic structure of the 5d states.3,4 The complete spatial isolation of the 5d metal ions in molecular complexes makes any magnetic interactions between the metal ions negligible, and therefore allowed the first experimental determination of a 5d spin and orbital momentum as well as the anisotropy of the 5d spin-orbit interaction. These results help to understand the overall magnetic behavior of 5d-based materials and to orient future design principles toward improved quantum magnets incorporating heavy transition elements.

Quantum Materials studied by inelastic neutron scattering


Niels Bohr Institute, University of Copenhagen

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Institute of Physics, Technical University of Denmark

plus numerous foreign collaborators

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Research in Quantum Materials deals with the quantum mechanical nature of matter. Besides being of fundamental interest, there is also a wish for unraveling the possibilities for application of quantum properties. One example that bridges basic science to technology is the study of quantum correlations – or entanglement – the understanding and control of which would be decisive for the construction of a quantum computer. The elusive high-temperature superconductors have a large potential for technological applications within energy, transport, and health - and magneto-electric – or multiferroic – materials have great potential for the IT industry. A key ingredient in quantum properties is the atomic spin, which also gives rise to magnetism. Therefore, the study of magnetism and quantum properties go hand in hand, lending neutron scattering a prime role in the study of quantum materials, since this probes both magnetic structure and dynamics with high resolution.

In this talk, I will present examples of the study of quantum materials taken place over the last decade in a collaboration between KU-NBI and DTU-Physics. I will give examples of the use of inelastic neutron scattering as a tool to understand fundamental quantum behaviour of the quasi-one-dimensional magnets CoCl$_2$ 2D$_2$O, the multiferroic material YMnO$_3$, and the high-temperature superconductor La$_{2-x}$Sr$_x$CuO$_{4+y}$. I will also briefly mention other present activities within magnetism and quantum material within the DANSCATT groups.

The Danish environment within magnetism and quantum materials are strongly engaged in the construction of the two Danish-led instruments for ESS: The diffractometer Heimdal and the spectrometer Bifrost. I will briefly discuss the plans for and status of these instruments. Finally, I will discuss which effect the instruments could have for the future of inelastic neutron studies of quantum materials.
The European XFEL

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The European X-ray Free Electron Laser is being commissioned in the spring of 2017 and will start first user operation in the fall. It will be the world’s first hard X-ray laser facility based on superconducting accelerator technology and will deliver an unprecedented X-ray beam to the user community with a high repetition rate. The outline of the facility is shown below. First users are expected to come in the fall of 2017 on the FXE instrument for ultra-fast x-ray spectroscopy and x-ray scattering and on the SPB/SFX instrument for diffractive imaging and structural determination for single particles, clusters and biomolecules. In 2018 four more instruments will be taken into operation covering a wide range of scientific fields. In the talk a full description of the facility will be given including a report of the status of the commissioning.
Mammalian crystalline proteins *in vivo* see the light

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It has been known since 1850 that human tissues harbouring parasitic infections also accumulate elongated hexagonal bi-pyramidal crystals in the extracellular spaces. These crystalline deposits grew to be widely known within the medical community as Charcot-Leyden crystals (CLC) and went on to be widely described in the lungs of asthmatics and patients with chronic rhinosinusitis. It took another 120 years before these crystals were biochemically defined as protein crystals made up of human Galectin-10 (also known as CLC protein) and a crystal structure based on recombinant human Galectin-10 ensued just before the turn of the new millennium. At around this time murine YM1, an unrelated protein to Galectin-10, had been shown to display similar auto-crystallization propensities in mice under conditions of inflammation. The structure of YM1 was subsequently determined based on crystals grown using YM1 isolated from murine peritoneal exudate cells.

However, two fundamental questions have remained unanswered: (i) What are the structures of human Galectin-10 and murine YM1 in crystals that grow *in vivo* under conditions of chronic inflammation, and (ii) What are the possible functions of such *in vivo* crystalline proteins.

My presentation will provide some initial answers to these questions as part of an *out-of-the-box* research undertaking that unites high-end X-ray crystallography and scattering methods, immunology, and university hospital operation theatres.
LINX - Linking Industry to Neutrons and X-rays

Erik Brok¹, Nicholas Skar-Gislinge¹, Martin Schmiele¹, Carsten Gundlach², Jacob Becker³, Mie Elholm Birkbak³, Andreas Bøggild⁴, Poul Nissen⁴ and Lise Arleth¹

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Neutron and X-ray scattering and imaging techniques have a huge potential for solving technological problems for the benefit of society. However, while these techniques have long been invaluable tools for academic research there is a lack of industrial use because most companies do not have the expertise to take advantage of the possibilities offered at modern large-scale facilities.

Following the large public investment in the European Spallation Source (ESS) the Danish government, through Innovation Fund Denmark, have invested in the LINX industry portal, with the goal of helping Danish industry benefit from ESS and the MaxIV synchrotron. LINX is a network of three Danish universities with expertise in neutron and X-ray methods, and presently 13 companies. The scientists help the industrial partners identify problems, which can be addressed with neutron and X-ray techniques, and with acquiring beam time, performing experiments, and analyzing data.

The goal is to establish a strong organization that can help the industry partners gain short and long-term technological advances, and develop new capabilities in neutron and X-ray techniques. Furthermore, it is our hope that an expanding user community will help push for developments in research infrastructure, instrumentation and sample environment to the benefit of the neutron and X-ray communities. In the presentation we will expand on the operation of the LINX organization and show examples of how we use X-ray and neutron methods to solve industrial problems.
DanMAX – The Danish Materials Science Beamline at MAX IV

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DanMAX will be a world-leading materials science beamline dedicated to in situ and operando experiments on real materials. The beamline will be built at the new MAX IV 3 GeV storage ring and will operate in the 15 - 35 keV range. It will have two end stations: one for full field imaging and one for powder X-ray diffraction. The combination of two related techniques will ensure cross talk between communities and likely seed new collaborations taking advantage of the high complementarity of the techniques. The beamline will be ready for first users in 2019.

Here we will present the DanMAX beamline and examples of the experiments envisioned at DanMAX. We will also present the recently started sample environment collaboration program where future users can collaborate on sample environment development. Finally, we will also give a status update on MAX IV and the other beamlines currently in construction and commissioning.
Soft Matter Science at ESS

Hanna Wacklin$^{1,2,*}$, Melissa Sharp$^1$, Andrew Jackson$^{1,2}$, Pascale Deen$^{1,3}$

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The ESS will be the world’s leading long pulse neutron source, with the first instrument expected to become operational in 2023. The suite of the first 15 instruments includes a number of beamlines for the study of soft condensed matter structure and dynamics. These instruments and their applications will all greatly benefit from the high flux and flexibility offered by the ESS long pulse structure and this will lead to at least an order of magnitude performance gain over existing instruments. This increase in performance will allow experiments that are currently at the edge of feasibility to become routine measurements, as well as new experiments to be developed. In this talk I will describe some current challenges and future developments enabled by the suite of reflectometers, SANS instruments, and neutron spectrometers, as well as the science support facilities at ESS.
<table>
<thead>
<tr>
<th>#</th>
<th>Presenting author</th>
<th>Affiliation</th>
<th>Poster title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Anders Filsæe Pedersen</td>
<td>DTU Physics</td>
<td>The fractional Fourier transform as a tool for CRL based X-ray microscopy</td>
</tr>
<tr>
<td>2</td>
<td>Anja Probst Larsen</td>
<td>University of Copenhagen</td>
<td>Identification and structure-function study of positive allosteric modulators of kainate receptors</td>
</tr>
<tr>
<td>3</td>
<td>Anna Zink Eikeland</td>
<td>Aarhus University</td>
<td>Enhancements of magnetic properties through morphology control of SrFe$<em>{12}$O$</em>{19}$ nanocrystallites</td>
</tr>
<tr>
<td>4</td>
<td>Beate Klösgen</td>
<td>University of Southern Denmark</td>
<td>A photoactive biomimetic membrane with binary behavior: 1. lipid dynamics</td>
</tr>
<tr>
<td>5</td>
<td>Bo Jakobsen</td>
<td>Roskilde University</td>
<td>Huginn: Peltier-based temperature controlled sample platforms for neutron scattering</td>
</tr>
<tr>
<td>6</td>
<td>Camilla Hjort Kronbo</td>
<td>Aarhus University</td>
<td>High Pressure Studies of SrGeO3 Polymorphs</td>
</tr>
<tr>
<td>7</td>
<td>Chen Gao</td>
<td>Aarhus University</td>
<td>Experimental Determination of the Valence Electron Density in a Dy-Based Single Molecule Magnet</td>
</tr>
<tr>
<td>8</td>
<td>Chen Shen</td>
<td>University of Southern Denmark</td>
<td>A photoactive biomimetic membrane with binary behavior: 2. membrane structure</td>
</tr>
<tr>
<td>9</td>
<td>Christian Henriksen</td>
<td>University of Southern Denmark</td>
<td>Phase transitions in hydroxyl iron phosphate cathodes for rechargeable lithium ion batteries</td>
</tr>
<tr>
<td>10</td>
<td>Christian Kolle Christensen</td>
<td>University of Southern Denmark</td>
<td>Operando scattering studies of insertion and extraction of Mg-ions in multilayered vanadium oxide nanotubes</td>
</tr>
<tr>
<td>11</td>
<td>Christian Lund Jakobsen</td>
<td>University of Southern Denmark</td>
<td>Morphology, crystalline structure and electrochemical properties of manganese phosphate cathodes for Li-ion batteries.</td>
</tr>
<tr>
<td>12</td>
<td>Christian Zeuthen</td>
<td>Aarhus University</td>
<td>Examining lead vacancies in lead chalcogenides and their effect on thermoelectric performance</td>
</tr>
<tr>
<td>13</td>
<td>Christina Wegeberg</td>
<td>University of Southern Denmark</td>
<td>In Situ Insight into Reversible O2 Gas-Solid Reactions</td>
</tr>
<tr>
<td>14</td>
<td>Christopher Rahl Andersen</td>
<td>University of Copenhagen</td>
<td>The H-T phase diagram of Ni$_3$TeO$_6$ with neutron diffraction on single crystals</td>
</tr>
<tr>
<td>15</td>
<td>Christopher Rahl Andersen</td>
<td>University of Copenhagen</td>
<td>Crystal and magnetic structures of Co$_2$MTeO$_6$ (M = Mg, Zn)</td>
</tr>
<tr>
<td>16</td>
<td>Claire Deville</td>
<td>Aarhus University</td>
<td>Coordination Polymers with Zinc(II) and Cobalt(II) Complexes as Chiral Molecular Bricks</td>
</tr>
<tr>
<td>17</td>
<td>Daniel Rasmussen</td>
<td>University of Copenhagen</td>
<td>Activating GluD2 Lurcher with D-serine by introducing GluN1 hinge and point mutation</td>
</tr>
<tr>
<td>18</td>
<td>Daniel Risskov Sørensen</td>
<td>University of Southern Denmark</td>
<td>Determination of phase transitions in Li-ion battery materials using synchrotron operando PXD</td>
</tr>
<tr>
<td>19</td>
<td>Dorthe Posselt</td>
<td>Roskilde University</td>
<td>Restructuring in block copolymer thin films: In situ GISAXS investigations during solvent vapor annealing</td>
</tr>
<tr>
<td>20</td>
<td>Drew Sheppard</td>
<td>Curtin University &amp; Aarhus University</td>
<td>Fluorine-Substituted Metal Hydrides for Thermal Energy Applications</td>
</tr>
<tr>
<td>No.</td>
<td>Name</td>
<td>Affiliation</td>
<td>Title</td>
</tr>
<tr>
<td>-----</td>
<td>-----------------------------</td>
<td>------------------------------------</td>
<td>----------------------------------------------------------------------</td>
</tr>
<tr>
<td>21</td>
<td>Elisabeth Grube</td>
<td>Aarhus University</td>
<td>Thermal energy storage materials in hydrogen storage</td>
</tr>
<tr>
<td>22</td>
<td>Ellen Hedegaard</td>
<td>Aarhus university</td>
<td>One Step Compositional Grading in the PbTe-SnTe system</td>
</tr>
<tr>
<td>23</td>
<td>Emil Damgaard-Møller</td>
<td>Aarhus University</td>
<td>Exchange mechanisms in a cobalt dimer</td>
</tr>
<tr>
<td>24</td>
<td>Emil Kjær</td>
<td>University of Copenhagen</td>
<td>Synthesis and characterization of CuCrO 2 delafossite nanocrystals</td>
</tr>
<tr>
<td>25</td>
<td>Emil Røssing Elholm</td>
<td>University of Copenhagen</td>
<td>Formation of NiO nanocrystal in organic solvents investigations of reaction mechanisms</td>
</tr>
<tr>
<td>26</td>
<td>Erik B Knudsen</td>
<td>NEXMAP, DTU Fysik</td>
<td>McXtrace 1.4, Newest features and Developments</td>
</tr>
<tr>
<td>27</td>
<td>Fabian Fantinelli Franco</td>
<td>Aarhus University</td>
<td>Cyclam-based Coordination Polymers for Gas Storage and Catalysis</td>
</tr>
<tr>
<td>28</td>
<td>Folmer Fredslund</td>
<td>University of Copenhagen</td>
<td>Structural characterization of the thermostable Bradyrhizobium japonicum d-sorbitol dehydrogenase</td>
</tr>
<tr>
<td>29</td>
<td>Frederik Gjerup</td>
<td>Aarhus University</td>
<td>In situ and ex situ annealing studies of hydrothermally synthesized SrFe12O19 nanoplatelets.</td>
</tr>
<tr>
<td>30</td>
<td>Giovanni Fevola</td>
<td>DTU</td>
<td>First Combined Scattering and Fluorescence Scanning Transmission Microscopy at the NanoMAX Beam Line at MAX IV</td>
</tr>
<tr>
<td>31</td>
<td>Heidi Asschenfeldt Ernst</td>
<td>University of Copenhagen</td>
<td>Structure of Myceliophthora thermophilia laccase</td>
</tr>
<tr>
<td>32</td>
<td>Henrik Jacobsen</td>
<td>Department of Physics, Oxford</td>
<td>Magnetic structure and dynamics of multiferroic CuO</td>
</tr>
<tr>
<td>33</td>
<td>Hidetaka Kasai</td>
<td>University of Tsukuba &amp; Aarhus</td>
<td>Charge Density Study of Thermoelectric ZnSb</td>
</tr>
<tr>
<td>34</td>
<td>Hugh Simons</td>
<td>DTU Physics</td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>Ida Gjerlevsen Nielsen</td>
<td>Aarhus University</td>
<td>Group 13 metal oxide formation, transformation and pre-nucleation: A study of the indium oxide system</td>
</tr>
<tr>
<td>36</td>
<td>Ida Haugaard Sørensen</td>
<td>Aarhus University</td>
<td>Synthesis and Characterization of Cyclam-based MOFs for Catalytic Applications</td>
</tr>
<tr>
<td>37</td>
<td>Jakob Grinderslev</td>
<td>Aarhus University</td>
<td>Synthesis of various metal borohydrides</td>
</tr>
<tr>
<td>38</td>
<td>Jakob Voldum Ahlburg</td>
<td>Aarhus University</td>
<td>Optimization of spring exchange coupled ferrites, studied by in situ neutron diffraction</td>
</tr>
<tr>
<td>39</td>
<td>Jakob Mikkelsen</td>
<td>Aarhus University</td>
<td>Pair distribution function analysis of Jahn-Teller fluctuation in copper Tutton salt</td>
</tr>
<tr>
<td>40</td>
<td>Jessica Lefevre</td>
<td>DTU</td>
<td>X-Ray microtomography and Raman studies on all-solid-state Li-S batteries built around LiBH4 solid electrolyte</td>
</tr>
<tr>
<td>41</td>
<td>Jette Katja Mathiesen</td>
<td>DTU Energy</td>
<td>Understanding the Mechanism of Sodium Insertion in Hard Carbon through Ex Situ Pair Distribution Function</td>
</tr>
<tr>
<td>42</td>
<td>Jette Oddershede</td>
<td>Xnovo Technology</td>
<td>Mapping grain morphology and orientation by Laboratory Diffraction Contrast Tomography</td>
</tr>
<tr>
<td>43</td>
<td>Joanna Symonowicz</td>
<td>University of Copenhagen</td>
<td>XRD total scattering of the CZTS nanoparticle absorber layer for the thin film solar cells</td>
</tr>
<tr>
<td>44</td>
<td>Jonas Okkels Birk</td>
<td>University of Copenhagen</td>
<td>Upgrade of the Primary spectrometer for RITA-II, PSI</td>
</tr>
<tr>
<td>Page</td>
<td>Author(s)</td>
<td>Institution(s)</td>
<td>Title</td>
</tr>
<tr>
<td>------</td>
<td>-----------</td>
<td>----------------</td>
<td>-------</td>
</tr>
<tr>
<td>45</td>
<td>Karl F. F. Fischer</td>
<td>Aarhus University</td>
<td>Anisotropic magnetoresistivity from van der Pauw measurements on LaSb2</td>
</tr>
<tr>
<td>46</td>
<td>Kasper Tolborg</td>
<td>Aarhus University</td>
<td>Core electron deformation in silicon from powder X-ray diffraction</td>
</tr>
<tr>
<td>47</td>
<td>Kasper Trans Møller</td>
<td>Aarhus University</td>
<td>A NaAlH4-Ca(BH4)2 Composite System for Hydrogen Storage</td>
</tr>
<tr>
<td>48</td>
<td>Kirstine Junker Dalggaard</td>
<td>Aarhus University</td>
<td>Core-shell TiO2@MoS2 for Li-ion Battery Anodes</td>
</tr>
<tr>
<td>49</td>
<td>Kirstine Louise Bendtsen</td>
<td>University of Copenhagen</td>
<td>Understanding protein-protein interactions relevant for neuronal spreading of alpha-synuclein</td>
</tr>
<tr>
<td>50</td>
<td>Lars Haahr Jepsen</td>
<td>Danish Technological Institute</td>
<td>The Danish Industrial Neutron Laboratory</td>
</tr>
<tr>
<td>51</td>
<td>Leila Lo Leggio</td>
<td>University of Copenhagen</td>
<td>Unliganded and substrate bound structures of the cellobiose polysaccharide active lytic polysaccharide monoxygenase LsAA9A at low pH</td>
</tr>
<tr>
<td>52</td>
<td>Lennard Krause</td>
<td>Aarhus University</td>
<td>SPAnPS – The Radiant Polymorphs</td>
</tr>
<tr>
<td>53</td>
<td>Line Abildgaard Ryberg</td>
<td>DTU Chemistry</td>
<td>Detemir-Albumin Interactions studied by small-angle X-ray scattering and in-silico modelling</td>
</tr>
<tr>
<td>54</td>
<td>Mads Folkjær</td>
<td>Aarhus University</td>
<td>Tailoring metal-organic frameworks: Connecting Co4O4 cubane complexes with the right organic linker</td>
</tr>
<tr>
<td>55</td>
<td>Maika Klemmer</td>
<td>Aarhus University</td>
<td>Hydrothermal liquefaction in a continuous flow reactor - what could possibly go wrong?</td>
</tr>
<tr>
<td>56</td>
<td>Martin Kjærulf Rasmussen</td>
<td>University of Copenhagen</td>
<td>Mesoporous silica as an oral adjuvant for hepatitis B vaccination</td>
</tr>
<tr>
<td>57</td>
<td>Martin Schmiele</td>
<td>University of Copenhagen</td>
<td>Small-angle scattering study on the structure of lecithin-stabilized tetracosane- water nanoemulsions and -suspensions</td>
</tr>
<tr>
<td>58</td>
<td>Martin Aaskov Karlsen</td>
<td>University of Southern Denmark</td>
<td>Structure and electrochemical performance of LiVPO4F as a cathode material</td>
</tr>
<tr>
<td>59</td>
<td>Mathias Helmsøe-Zinck</td>
<td>University of Copenhagen</td>
<td>Synthesis and characterization of MoO2 nanoparticles</td>
</tr>
<tr>
<td>60</td>
<td>Mathias Hvid</td>
<td>Aarhus University</td>
<td>Modification of light absorption properties of 2D thiostannates</td>
</tr>
<tr>
<td>61</td>
<td>Mathias Jørgensen</td>
<td>Aarhus University</td>
<td>Halogenated sodium-closo-dodecaboranes as solid-state ion conductors</td>
</tr>
<tr>
<td>62</td>
<td>Mathias Mørch</td>
<td>Aarhus University</td>
<td>Effect of precursor choice and sintering conditions for SrZn2-W hexaferrite formation</td>
</tr>
<tr>
<td>63</td>
<td>Mattia Sist</td>
<td>Aarhus University</td>
<td>GeTe: focus on the high-temperature phase transition through high resolution XRPD</td>
</tr>
<tr>
<td>64</td>
<td>Mikkel Juelsholt</td>
<td>University of Copenhagen</td>
<td>Temperature dependent intrinsic defects in tungsten oxide nanorods</td>
</tr>
<tr>
<td>65</td>
<td>Nicholai Daugaard Jensen</td>
<td>SDU</td>
<td>Study of layered double hydroxides intercalated with para-aminosalicylate</td>
</tr>
<tr>
<td>66</td>
<td>Nicolai Ytterdal Juul</td>
<td>DTU</td>
<td>Deformation Mechanics of 316 Stainless Steel on the Level of Individual Grains Observed In-situ with 3DXRD Microscopy and Compared to Crystal Plasticity Simulations</td>
</tr>
<tr>
<td>67</td>
<td>Niklas Durhuus Vichairat</td>
<td>Aarhus University</td>
<td>Synthesis and reduction of iron oxides to alpha-iron</td>
</tr>
<tr>
<td>68</td>
<td>Nikolaj Roth</td>
<td>Aarhus University</td>
<td>Magnetic short range ordering in frustrated magnets</td>
</tr>
<tr>
<td>69</td>
<td>Paolo Lamagni</td>
<td>Aarhus University</td>
<td>Direct growth of HKUST-1/graphene composite films on glassy carbon</td>
</tr>
<tr>
<td>70</td>
<td>Per Sigaard Christensen</td>
<td>Danish Technological Institute</td>
<td>Controlling residual stresses in industrial components</td>
</tr>
<tr>
<td>71</td>
<td>Peter Willendrup</td>
<td>DTU Physics</td>
<td>New developments in McStas</td>
</tr>
<tr>
<td>#</td>
<td>Author</td>
<td>Affiliation</td>
<td>Research Title</td>
</tr>
<tr>
<td>----</td>
<td>-------------------------</td>
<td>--------------------------------------</td>
<td>-------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>72</td>
<td>Pia Jensen Ray</td>
<td>University of Copenhagen</td>
<td>The e-neutrons.org platform - developing a platform for teaching of neutron scattering using blended learning</td>
</tr>
<tr>
<td>73</td>
<td>Pia Jensen Ray</td>
<td>University of Copenhagen</td>
<td>Complex superstructures in the co-doped high-Tc superconductor La(2-x)Sr(x)CuO(4+y), observed with both neutrons and X-rays and &quot;The e-neutrons.org platform - developing a platform for teaching of neutron scattering using blended learning&quot;</td>
</tr>
<tr>
<td>74</td>
<td>Ralph Bolanz</td>
<td>Friedrich-Schiller University Jena</td>
<td>Investigation of close-range order phenomena during homo- and heterovalent substitution reactions – A summary of X-ray-absorption spectroscopy studies</td>
</tr>
<tr>
<td>75</td>
<td>Sara Ane Zachhau</td>
<td>University of Southern Denmark</td>
<td>Solid state NMR investigations of North Sea Chalk</td>
</tr>
<tr>
<td>76</td>
<td>Simon Rosenqvist Larsen</td>
<td>University of Copenhagen</td>
<td>Dynamical analysis of Ciprofloxacin intercalated in fluorohectorite</td>
</tr>
<tr>
<td>77</td>
<td>Simone M. Kevy</td>
<td>Aarhus University</td>
<td>Superconducting Topological Materials by Doping of Bi2Se3</td>
</tr>
<tr>
<td>78</td>
<td>Sonja Holm</td>
<td>Aarhus University</td>
<td>HEIMDAL: A thermal neutron powder diffractometer with high and flexible resolution combined with SANS and neutron imaging – Designed for materials science studies at the European Spallation Source</td>
</tr>
<tr>
<td>79</td>
<td>Sonja Rosenlund Ahl</td>
<td>DTU Physics</td>
<td>Recovery and recrystallization studied by dark field x-ray microscopy</td>
</tr>
<tr>
<td>80</td>
<td>Sotirios Magkos</td>
<td>University of Copenhagen</td>
<td>Neutron and X-ray tomography of charred cereal grains from archaeological site in southern Sweden</td>
</tr>
<tr>
<td>81</td>
<td>Steffen Riis Højbjerg Jensen</td>
<td>Aarhus university</td>
<td>Investigation of NH3 containing alanates</td>
</tr>
<tr>
<td>82</td>
<td>Saara Laulumaa</td>
<td>University of Copenhagen</td>
<td>Exploring the ligand binding site of ionotropic glutamate receptor using neutron crystallography.</td>
</tr>
<tr>
<td>83</td>
<td>Tiago Ramos</td>
<td>DTU</td>
<td>Flexible 3D tomographic alignment and reconstruction of phase-contrast data</td>
</tr>
<tr>
<td>84</td>
<td>Tim Tejsner</td>
<td>University of Copenhagen &amp; Institut Laue-Langevin</td>
<td>Phonon contributions to high-temperature superconductivity</td>
</tr>
<tr>
<td>85</td>
<td>Troels Lindahl Christiansen</td>
<td>University of Copenhagen</td>
<td>Atomic structure in molybdenum oxide nanoparticles</td>
</tr>
<tr>
<td>86</td>
<td>Ursula Bengaard Hansen</td>
<td>University of Copenhagen</td>
<td>An optional focusing SELENE extension to conventional neutron guides: A case study for the ESS instrument BIFROST</td>
</tr>
<tr>
<td>87</td>
<td>Yigang Yan</td>
<td>Aarhus University</td>
<td>Beyond Hydrogen Storage, Complex Hydrides as Solid Electrolyte for Battery</td>
</tr>
</tbody>
</table>
**List of participants** – by affiliation and first name

<table>
<thead>
<tr>
<th>Name</th>
<th>Affiliation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anna Zink Eikeland</td>
<td>Aarhus University</td>
</tr>
<tr>
<td>Bo Brummerstedt Iversen</td>
<td>Aarhus University</td>
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<tr>
<td>Camilla Hjort Kronbo</td>
<td>Aarhus University</td>
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<td>Casper Welzel Andersen</td>
<td>Aarhus University</td>
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<td>Chen Gao</td>
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<td>Claire Deville</td>
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<td>Drew Sheppard</td>
<td>Curtin University &amp; Aarhus University</td>
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<td>Elisabeth Grube</td>
<td>Aarhus University</td>
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<td>Ellen Hedegaard</td>
<td>Aarhus University</td>
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<td>Emil Damgaard-Møller</td>
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<td>Fabiane Fantinelli Franco</td>
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<td>Frederik Gjørup</td>
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<td>Hao Tang</td>
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<td>Hidetaka Kasai</td>
<td>University of Tsukuba &amp; Aarhus University</td>
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<td>Ida Gjerlevsen Nielsen</td>
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<td>Ida Haugaard Sørensen</td>
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<td>Jacob Becker</td>
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<td>Jakob Grinderslev</td>
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<td>Jennifer Hoelscher</td>
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<td>Mads Ry Jørgensen</td>
<td>Aarhus University &amp; MAX IV</td>
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<td>Maika Klemmer</td>
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<td>Mathilde Nygaard</td>
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<td>Matthew Craven</td>
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<td>Mattia Sist</td>
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<tr>
<td>Mie Birkbak</td>
<td>Aarhus University &amp; LINX</td>
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<tr>
<td>Mogens Christensen</td>
<td>Aarhus University</td>
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<tr>
<td>Morten Bormann Nielsen</td>
<td>Aarhus University</td>
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<tr>
<td>Niklas Durhuus Vichairat</td>
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<td>Nikolaj Roth</td>
<td>Aarhus University</td>
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<td>Nina Lock</td>
<td>Aarhus University</td>
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<td>Paolo Lamagni</td>
<td>Aarhus University</td>
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<tr>
<td>Simone M. Kevy</td>
<td>Aarhus University</td>
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Poster abstracts
The fractional Fourier transform as a tool for CRL based X-ray microscopy

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The fractional Fourier transform (FrFT) was first introduced as an extension of the well-known Fourier transform\textsuperscript{1,2}, allowing one to perform rotations in phase space of arbitrary degree. The Fresnel diffraction expression may be reformulated in terms of the FrFT, and can be used to simulate the free space wave front propagation much like traditional Fourier optics\textsuperscript{3,4}. For the traditional Fourier optics a free space propagation may be interpreted as a normal Fourier transform transforming the wave front on one curved surface to another, using scaled variables. The radius of curvature of the origin and destination surfaces and the variable scaling are all defined by the physical dimensions. For the fractional Fourier optics the extra degree of freedom provided by the degree of the Fourier transform allows infinite choices of radius of curvature and variable scaling of the origin plane. This combined with the additive nature of the transform order allows one to propagate the wave front through any number of lenses or planes in a single transform, as long as they do not have any attenuation. This approach allows us to quickly and efficiently simulate wave front propagation through highly complex setups used at beam lines, including compound refractive lenses (CRL). As an example we quantify the resolution (width of the point spread function) of a setup combining a CRL objective with a slit in the back focal plane. We find that a full wave front simulation is required to estimate the resolution in the regime where the resolution is not severely restricted by one of the two components. Furthermore, the speed of the simulation allows us to perform partially coherent (both temporal and spatial) simulations quickly.

Figure 1: By using the fractional Fourier transform it is possible to perform a full wave front propagation simulation through a CRL with N lenses, through a slit in the back focal plane, and to the detector in the image plane in just 3 steps. In this approach an effective aperture is used to model the absorption inside the CRL.

Identification and structure-function study of positive allosteric modulators of kainate receptors

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Kainate receptors (KARs) consist of a class of ionotropic glutamate receptors, which exert diverse pre- and postsynaptic functions through complex signaling regulating the activity of neural circuits. Whereas numerous small-molecule positive allosteric modulators of the ligand-binding domain (LBD) of AMPA receptors have been reported, no such ligands are available for KARs. In this study, we have investigated the ability of three benzothiadiazine-based modulators to potentiate glutamate-evoked currents at recombinantly expressed KARs.

Figure 1. Chemical structures of the three benzothiadiazine-based positive allosteric modulators.

BPAM344 potentiated glutamate-evoked currents of KAR subunit GluK2a 21-fold at the highest concentration tested (200 µM), with an EC₅₀ of 79 µM, and markedly decreased desensitization kinetics from 5.5 to 775 ms. BPAM521 potentiated the recorded peak current amplitude of GluK2a 12-fold at a concentration of 300 µM with an EC₅₀ value of 159 µM, whereas no potentiation of the glutamate-evoked response was observed for BPAM121 at the highest concentration of modulator tested (300 µM). BPAM344 (100 µM) also potentiated the peak current amplitude of KAR subunits GluK3a (59-fold), GluK2a (15-fold), GluK1b (5-fold), as well as the AMPA receptor subunit GluA1i (5-fold). X-ray structures of the three modulators in complex with the GluK1 LBD were determined, locating two modulator-binding sites at the GluK1 LBD dimer interface. In conclusion, this study opens for the design of new positive allosteric modulators selective for KARs, which will be of great interest for further investigating the function of KARs in vivo and may prove useful to pharmacologically control the activity of neuronal networks.

2 Pohlsgaard et al., 2011. Lessons from more than 80 structures of the Glu2 ligand-binding domain in complex with agonists, antagonists and allosteric modulators. Neuropharmacology 60, 135-150.
Enhancements of magnetic properties through morphology control of SrFe$_{12}$O$_{19}$ nanocrystallites

Anna Zink Eikeland, Marian Stingaciu, Mogens Christensen
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Permanent magnets have great importance for many technological applications. Today, researchers focus at making high performance and cheap magnets without rare earth elements. Strontium hexaferrite, SrFe$_{12}$O$_{19}$, is an important permanent magnet because of its large anisotropy, good performance/cost ratio, and high stability.

SrFe$_{12}$O$_{19}$ can be made as magnetic single domain nanoparticles allowing enhancement of the magnetic performance. Among others factors the magnetic performance depends, on the morphology, which influences the demagnetisation factor and particle’s ability to align. It is important that the particles are easy to align since they must be compacted into a bulk material for real world applications. The morphology is very dependent on the synthesis method: hydrothermal synthesis forms large plate-like nanoparticles, whereas a conventional sol-gel synthesis results in small square-like intergrown particles. A modified sol-gel synthesis makes the particles small plate-like as a cross between the autoclave and conventional sol-gel syntheses.

In present work the SrFe$_{12}$O$_{19}$ powder was made using the three above mentioned synthesis methods to investigate how the magnetic properties depend on morphology. Furthermore, the particles were compacted into dense pellets using a Spark Plasma Sintering system, the study reveal how the particles align within the pellet and how is effect the magnet performance.

It was found that the autoclave compacted powder become highly aligned resulting in an energy product of 31 kJ/m$^3$ which is 244 % larger than the energy product obtained from the powder. For the conventional sol-gel the difference is only 24 % indicating that the particles aren’t well aligned which can be confirmed from X-ray pole figure measurements. The magnetic performance of the modified sol-gel synthesised SrFe$_{12}$O$_{19}$ is improved more than 100 % to 20 kJ/m$^3$ and the crystallites are better aligned than the conventional sol-gel. However, compared to the autoclave synthesised SrFe$_{12}$O$_{19}$, the crystallites are still poor aligned but may be improved by changing the compacting parameters.
A photoactive biomimetic membrane with binary behavior:

1. lipid dynamics

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Cholesterol (chol) is a natural membrane compound. Its photosensitive variety azobenzene-cholesterol (azo-chol) exhibits a reversible change of the conformation in the headgroup region upon illumination (365nm: trans to cis; 455nm: cis to trans). We present a membrane system that actively responds to external illumination by use of the photoswitching of azo-chol. The model system consists of a 1-palmitoyl-2-oleoyl-sn-phosphatidyl-choline (POPC) matrix with embedded azo-chol (95:5, mol/mol) in either of the two conformational states. Its lipid dynamics was studied by quasielastic neutron scattering (QENS). A pure POPC membrane and also a POPC membrane with chol were used as control systems. The results report on several dynamic modes. These are intramolecular motions of the protons within the POPC chains and two motion modes of the whole molecule. These are a hop diffusion and a slow motion. The intramolecular motion and the hop diffusion are modified by the presence of azo-chol in the host membrane as compared with the control systems, but do not depend on the isomerization state. The slow mode is though sensitive to the isomerization. The use of azo-chol thus turns the membrane into an active system: its dynamics and most probably also the structure can be tuned between two binary states by illumination.
Huginn: Peltier-based temperature controlled sample platforms for neutron scattering

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The main goal of the RUC/ESS Huginn project is to utilize Peltier elements for improving temperature stability and decreases thermalization time compared to conventional sample environments used in neutron scattering.

The ability to change temperature fast, has the general advantage that more temperatures can be investigated in a given time interval. This becomes increasingly important at the ESS with the greater neutron brightness and therefore shorter measurement time.

Furthermore, the ability for fast temperature change of the sample will open new possibilities such as: Studies involving time resolved measurements which are performed on processes that start when the sample is moved to a given temperature, i.e. physical aging, crystallization, water uptake or chemical reactions. It also becomes possible to access new meta-stable phases of the investigated substances. Finally, studies of glass-forming liquids and other system with strong temperature dependence will benefit greatly by the increased stability and precision of the sample temperature.

We present design and prototype tests of two Peltier element based sample holders:

- A sub-cryostat insert for top loading cryostats, for use with general purpose sample cells.
- A multiple temperature SANS sample environment.

The sub-cryostat insert has the following key performance parameters:

- Very fast temperature changes, typically a few minutes for 10 - 20K temperature changes.
- Improved temperature stability compared to the main-cryostat.
- A working temperature range from 100K to 370 K.

The multiple temperature SANS sample environment has the following performance parameters:

- Individual cuvettes can have a temperature difference from the base temperature of 30 K.
- Temperature changes of up to 30K can be achieved within a few minutes.
- The temperature is stabilized within 10-100 mK.

Altogether we demonstrate that Peltier element based sample environments is able to decreases thermalization time and improve temperature stability. The two sample environments presented will be available at the ESS, and can easily be adapted to other facilities.
High Pressure Studies of SrGeO₃ Polymorphs

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The high pressure structural behaviors of SrGeO₃ polymorphs have been investigated in diamond anvil cells (DACs) using synchrotron powder X-ray diffraction (PXRD), single crystal diffraction (SC-XRD) and in-situ high-temperature high-pressure PXRD in a large volume press (LVP). The thermodynamically stable pseudo-wollastonite SrGeO₃ (space group C2/c) was synthesized at ambient pressure and subsequently a fraction was transformed in a LVP to the cubic perovskite SrGeO₃ (space group Pm-3m). Upon compression in a DAC at room temperature, Rietveld refinements showed that the perovskite retains the cubic symmetry up to the highest pressure of 45 GPa. For the pseudo-wollastonite SrGeO₃ it was seen that the sample gradually turns amorphous above 11 GPa as shown in Figure 1. Fitting a 3rd order Birch-Murnaghan equation of state to the obtained P-V data for the two polymorphs yielded bulk moduli of $K_0 = 47(4)$ GPa and $K_0 = 194(3)$ GPa for the pseudo-wollastonite and perovskite polymorphs, respectively, showing the perovskite structure is much less compressible.

Previous studies of pseudo-wollastonite SrGeO₃ at high pressure and high temperature have shown a phase transition at 1 GPa, but the structure of the high pressure phase was not solved at the time¹. To investigate the structural details of a subtle transition at 1 GPa and room temperature for pseudo-wollastonite SrGeO₃ we used SC-XRD in a DAC up to 10 GPa. The initial symmetry of the crystal was retained up to 10 GPa with no indication of a phase transition. In recent in-situ studies using high-temperature high-pressure PXRD at ESRF ID06, we extended the studies to include high temperature. The ongoing data analysis suggests several extensions to P-T phase diagram of SrGeO₃.

Experimental Determination of the Valence Electron Density in a Dy-Based Single Molecule Magnet

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Electrostatic charge repulsion model has been set up and used to predict the easy axis and crystal field splitting of lanthanide complexes [1-3]. However, direct measurement of the f-electrons of Dy(III) ion is still a question. Here we studied the electron density in a Dysprosium single-ion magnet, Dy(DBM)(BPY) (1) by refinement of single crystal X-ray data from synchrotrons at 20K. Topological analysis showed closed shell interactions between Dy ion and the coordination atoms. Mapping and analysis of the shape of Dy valence shell indicated asphericity and oblate shape of the f-electron states. 3D isosurfaces of charge density are drawn, and ellipsoid fitting lead to the estimation of the magnetic anisotropic easy axis direction. The result show that the valence shell charge density of 1 has oblate shape and the predicted direction has 35° difference from the ab initio result. This is the first time to measure the distribution of valence shell charge density in lanthanide single ion magnet. The study gives experimental evidence of the relationship between structure and magnetic anisotropy in lanthanide compounds.

Figure. Shortest ellipsoid axis from surface fitting of valence charge density isosurface (red), and the easy axis from MOLCAS calculations (green)


Acknowledgements -

This work is supported by Danish National Research Foundation (DNRF-93) and Danscatt.
Cholesterol (chol) is a natural membrane compound. Its photosensitive variety azobenzene-cholesterol (azo-chol) exhibits a reversible change of the conformation in the headgroup region upon illumination (365nm: trans to cis; 455nm: cis to trans). We present a membrane system that actively responds to external illumination by use of the photoswitching of azo-chol. The model system consists of a 1,2-dipalmitoyl-sn-phosphatidyl-choline (DPPC) monolayer with embedded azo-chol (85:15, mol/mol) in either of the two conformational states. Its lateral structure was studied by grazing incidence X-ray diffraction (GIXD). A pure DPPC monolayer and also a DPPC monolayer with chol were used as control systems. The results report on the symmetry of the 2D-lattice and on its dimensions. The tilt angle of the lipid chains was calculated from the lattice parameters and the geometrical boundary conditions. Below a surface pressure of 40mN/m, the lattice dimensions and especially the chain tilting are modified when azo-chol in the host membrane is in all-trans state as compared to the control systems. With all-cis azo-chol, the monolayer is disordered. The use of azo-chol thus turns the membrane into an active system: its lateral structure can be tuned between two binary states by illumination.
Phase transitions in hydroxyl iron phosphate cathodes for rechargeable lithium ion batteries

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Electrical energy storage of renewable energy is widely recognized as a potential substitution for fossil fuels. In the realization of this scenario, lithium ion batteries hold great potential to become an essential mean of storage. However, for grid-scale (MWh) storage of electrical energy it is necessary for the battery to be cheap, safe, long lasting and have a high rate capability. The latter means that the battery is capable of going through a rapid discharge without significant decrease in the effective delivered capacity.\textsuperscript{1} To fulfill this purpose phospho-olivine materials, LiMPO\textsubscript{4} (M = Fe, Mn, Ni, Co) are attractive cathode materials for Li-ion batteries, which besides high rate capability also have high theoretical capacity, high lithium intercalation voltage, cheap production cost, and high stability. Lithium iron phosphate is already widely used with great success in electrical vehicles and portable power tools.\textsuperscript{2} However, there is still great interest in improving the rate capability further to increase the effective battery capacity, which especially is of great economic interest for large grid-scale battery applications.

Inclusion of other electronegative moieties, i.e. F\textsuperscript{−} or OH\textsuperscript{−}, into the framework of the olivine structure also carries potential to improve the rate capability compared to LiFePO\textsubscript{4} due to inductive effects lowering the activation energy of the transition metal oxidation and hence of the lithium release from the host structure.\textsuperscript{3} Furthermore, these compounds may exhibit phase transformations with high degree of solid solution formation and coherent behavior at the atomic lattice scale, which is a criteria for obtaining high rate capabilities.

Here we have focused on iron hydroxyphosphates of general composition Fe\textsubscript{2−x}PO\textsubscript{4}OH\textsubscript{3−3x} for which little information on their phase transitions during battery charge and discharge is known. These have been investigated using operando synchrotron powder X-ray diffraction during battery operation.

References:
Operando scattering studies of insertion and extraction of Mg-ions in multilayered vanadium oxide nanotubes

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Exchanging the active specie, Li⁺ in Li-ion batteries by multivalent, abundant and cheap cations, such as Mg^{2+}, are projected to boost the energy density and lower the cost per kilo-watt-hour significantly, making the Mg-ion battery technology a promising candidate for one of the battery technologies of the future.¹ The increase in energy density is i.a. a result of the divalent Mg^{2+} carrying twice as much charge as the monovalent Li⁺. However, the higher charge also poses a problem as it significantly increases the charge density of the ion, which results in stronger interactions with the host lattice of the electrodes and hampers facile ion transport. Therefore, development of novel electrode materials for effective Mg-ion storage is a vital step for the realization of this battery technology.² One way of enhancing electrode materials is nanosizing.³ Developments in the control of nanostructured morphologies have led to more advanced materials, and recently vanadium oxide nanotubes (VOₓ-NT) were shown to perform well as a cathode material for Mg-ion batteries.⁴ The VOₓ-NTs consists of multiwalled scrolls of crystalline VOₓ layers with approximate composition V₇O₁₆ and primary amines in between the layers acting as spacer molecules, which can be exchanged with metal cations with a subsequent change in layer spacing.⁵ In this study we have synthesized a series of VOₓ-NTs with varying spacer molecules. The mechanism for Mg-intercalation and deintercalation was studied by operando X-ray scattering measured during battery operation.

References:

Morphology, crystalline structure and electrochemical properties of manganese phosphate cathodes for Li-ion batteries

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Interest in rechargeable batteries continues to grow and in recent years there has been special focus on developing cheaper, safer and more stable batteries. In this respect, one of the most promising cathode materials in the battery industry is olivine LiFePO4 (LFP). However, during charge and discharge of the battery LFP cathodes exhibit of a two-phase behavior, i.e. direct transformation between LFP and FP. Such transitions can result in slow kinetics. Recent studies show that such two phase transitions can be prevented is by incorporating manganese into the cathode material, such that a Mn-Fe solid solution will be obtained.

In this work, hydrothermal synthesis has been applied for synthesis of phosphate based cathode materials). Especially a series of manganese-phosphate based samples were found to exhibit very good electrochemical performance, which were initially subscribed to the only well-crystalline compound MnPO4·nH2O in the samples. However, operando synchrotron powder X-ray diffraction measured during battery charge and discharge revealed that the electrochemical active phase is not the MnPO4·nH2O but instead what is likely a nanocrystalline phase. This phase is further characterized using scanning electron microscopy and thermogravimetric analysis.

Top: Operando Synchrotron Powder X-ray Diffraction measured during battery charge and discharge using a cathode of MnPO4·nH2O and an unknown active nanocrystalline compound. The blue ticks to the left are the peak positions for MnPO4·nH2O. Bottom: Simultaneously collected electrochemical data for the galvanostatic charge and discharge.
Examining lead vacancies in lead chalcogenides and their effect on thermoelectric performance

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Lead chalcogenide thermoelectric systems have been known since the 1950s and have been used in several applications since. Extensive theoretical and experimental research on the lead chalcogenides has been conducted to understand the nature of their high thermoelectric performance, which makes lead chalcogenides one of the most studied thermoelectric materials. The reported carrier concentrations of the pure substance may deviate across samples, sometimes even to the point of disagreement between the majority carrier type. The source of this discrepancy has not been considered in detail previously, but it has been speculated to be an effect of small changes in stoichiometry. [1]

A recent study by Christensen et. al.[2] reported cation vacancies in all of the lead chalcogenides investigated by Powder Crystal X-ray Diffraction (PXRD); most pronounced in the lead sulfide system, where two different samples had 2-4% and 4-8% lead vacancies. Meanwhile, the lead telluride and lead selenide systems were closer to the nominal stoichiometry, with only 1-2% lead vacancies. Traditionally, the lead chalcogenides are reported to be a line-phase with negligible deviation from stoichiometry [3]. The recently discovered deviation could explain the differences in material’s properties observed in literature, but so far this deviation has only been shown by Rietveld refinement on PXRD.

This presentation will aim to reveal subtle changes in stoichiometry of lead chalcogenides by using several different material characterization methods such as synchrotron Powder and Single Crystal X-ray Diffraction (PXRD/SCXRD), Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), Energy-dispersive X-ray Spectroscopy (EDX), among others. In addition, these subtle changes will be linked to thermoelectric properties by measuring the Seebeck coefficient, thermal transport, resistivity and charge carrier concentration of a range of samples to reveal the effect of lead deficiency.

Figure 1: PbTe crystals growing from a pellet.

In Situ Insight into Reversible O₂ Gas-Solid Reactions

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Non-porous crystalline solids containing a series of cationic tetracobalt complexes reversibly, selectively and stoichiometrically chemisorb dioxygen in temperature/O₂ partial pressure induced processes involving the oxidation of cobalt with concurrent reduction of two equivalents of sorbed O₂ to form μ-η¹, η²-peroxide ligands [1]. The attenuation of O₂ affinity by the introduction of electron withdrawing or electron donating substituents into the supporting ligand framework, otherwise dominant in solution [2] is overridden in the crystalline state. Here O₂ affinity is tuned predominantly by phase and a two-step gas sorption isotherm is apparent [1]. By following in situ reversible single-crystal to single-crystal (SCSC) transformations [3] using a gas-crystal cell and synchrotron X-ray radiation we can show that two distinctive channels through the crystalline solids are operative under sorption and desorption processes [4]. The reaction proceeds via a semi-oxy form that we had been seeking for some time, and the results have enabled an updated hypothesis that the transient conduit for O₂ escapes from an oxy crystal, albeit a counter anion obstacle race is lined by stacks of one end of the molecule. This is contrary to our original hypothesis [1] that communication between the binding sites caused an allostery or anti-cooperative effect: Crystal structure overrides molecule tweaking.

Figure. Top: tetracobalt complex in oxy, semi-oxy and deoxy form. Bottom: crystals in oxy (left) and (almost) deoxy form (right).

References:
The $H$-$T$ phase diagram of Ni$_3$TeO$_6$ with neutron diffraction on single crystals

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Multiferroic compounds with the coexistence of magnetic and ferroelectric orders are well known from few transition metal oxides with perovskite crystal structures. Recently, a new family of multiferroic oxides with other crystal structures has been found: $M_3$TeO$_6$ ($M =$ Co, Mn, Ni).

Ni$_3$TeO$_6$ has shown interesting multiferroic behaviour with its non-centrosymmetric structure and collinear antiferromagnetic order. A field induced second order spin flop transition has formerly been reported with a non-hysteric colossal magneto-electric coupling.

We have performed, for the first time, neutron diffraction measurements on Ni$_3$TeO$_6$ single crystals (figure 1) in high magnetic fields. The second order spin flop transition from earlier reports is not observed, but a different phase transition is found from a commensurate antiferromagnetic peak to an incommensurate peak. The $H$-$T$ phase diagram is mapped and we observe a rise in the critical field close to 20 K, where the incommensurate peak also starts to drift.

The present findings invite to a discussion of not only the magnetic system, but also the mechanism behind the multiferroicity and the magneto-electric coupling.

Figure 1: Picture of a single crystal with the hexagonal crystal lattices illustrated. The experiment consisted of a number of co-aligned single crystals, and the results will be presented at the conference.
Crystal and magnetic structures of $\text{Co}_2\text{MTeO}_6$ ($\text{M} = \text{Mg, Zn}$)

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Multiferroic compounds with the coexistence of magnetic and ferroelectric orders are well known from transition metal oxides with perovskite crystal structures. Recently, a new family of multiferroic oxides with other crystal structures has been found: $\text{M}_3\text{TeO}_6$ ($\text{M} = \text{Co, Mn, Ni}$).

$\text{Co}_3\text{TeO}_6$ has shown a complex magnetic ordering with several positions for the magnetic cations. This study presents the results of substituting some of these magnetic cations with non-magnetic cations, which has not been reported earlier. The crystallographic and magnetic structures of $\text{Co}_2\text{MTeO}_6$ ($\text{M} = \text{Mg, Zn}$) have been studied with polycrystalline samples using neutron powder diffraction (NPD), magnetization and specific heat measurements.

At room temperature, the crystal structure of $\text{Co}_2\text{ZnTeO}_6$ is similar to the cryolite crystal structure of $\text{Co}_3\text{TeO}_6$, being monoclinic space group $C2/c$, which remains stable down to 2 K. A second order phase transition is observed below $T_N = 13.2(3)$ K, which has been found to originate from antiferromagnetic interactions with an effective magnetic moment of $\mu_{\text{eff}} = 3.49 \mu_B/\text{Co}$ corresponding to high spin $\text{Co}^{2+}$ ($S = 3/2$). The magnetic structure cannot be determined from the NPD data.

At room temperature, the crystal structure of $\text{Co}_2\text{MgTeO}_6$ is a corundum crystal structure similar to that of $\text{Mn}_3\text{TeO}_6$ and $\text{Mg}_3\text{TeO}_6$, being trigonal space group $R3$, which remains stable down to 2 K. It has an antiferromagnetic structure characterized by a propagation vector of $k = (0, 0, \frac{1}{2})$ below $T_N = 29.06(10)$ K with a second order phase transition. The magnetic moments are oriented along the $a$-$b$-plane with $120^\circ$ between the nearest neighbors in the plane. The effective magnetic moment has been found to be $\mu_{\text{eff}} = 3.97 \mu_B/\text{Co}$ corresponding to high spin $\text{Co}^{2+}$ ($S = 3/2$).
Coordination Polymers with Zinc(II) and Cobalt(II) Complexes as Chiral Molecular Bricks

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Chiral metal-organic frameworks (MOFs) have retained increasing attention during the past ten years. The applications of chiral MOFs are typically in enantiomeric separation[1] and heterogeneous catalysis,[2] but more exotic uses such as X-ray analysis of non-crystallisable organic molecules have also been reported.[3]

In presence of a base, the bis-benzimidazole derivative of S-malic acid forms mononuclear zinc(II) and tetranuclear cobalt(II) complexes, which create homochiral polymeric chains in combination with dicarboxylate linkers. The structure of the zinc polymer (Fig. 1a) shows the chains to be slightly helical (P-type) and interact four by four through π-stacking and hydrogen bonding interactions around a 4₁ screw axes. In the cobalt(II) structures, the tetranuclear cubane-like units[4] are linked together by diagonally bridging linear linkers (Fig. 1b). Four types of crystals were obtained from the same sample differing in morphology and hue. Two of these structures are polymorphs while the two others slightly differ in composition. The salient point is that the polymeric chains of cubane-like complexes are present as the main component in all the structures.

Figure 1. X-ray crystal structures of polymeric chains of zinc(II) (a) and cobalt(II) (b) complexes.

Activating GluD2 lurcher with D-serine by introducing GluN1 hinge and point mutation

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Excitatory neurotransmission in the brain is mediated by the ionotropic glutamate receptors (iGluRs), classified as NMDA, AMPA, kainate and delta (GluD1 and GluD2), among which the delta receptors are by far the least understood 1. The GluD2 receptors are located in the purkinji cells, inside the cerebellum. GluD2 are involved in long-term depression along with synaptogenesis, and has been associated with synaptic plasticity and diseases as ataxia 2. In contrary to the other iGluRs, GluD2 cannot bind glutamate, and so far, no ligand is known that can activate the ion channel.

Figure 1 represents the ligand D-serine that is known to bind to GluD2 3.

The group of Prof. Kastrup showed that D-serine binds to the ligand-binding domain (LBD) of wildtype GluD2 and that it inactivates the spontaneously active GluD2 containing the lurcher mutation (EC 50 182 µM). Replacing the hinge region from GluD2 with the one from GluN1 changes the function and binding of D-serine. The same study showed that mutating a tyrosine at position 770 into a phenylalanine increased the binding affinity 7-fold (Kd = 117 µM vs. 809 µM) 1. As the insertion of the GluN1 hinge region and the Y770F point mutation was done separately, the aim of the project is to insert both the GluN1 hinge region and the Y770F point mutation into the GluD2-LBD, and analyze the changes in binding affinity by isothermal titration calorimetry (ITC). The current state of the project is purification of the GluD2-LBD-Y770F-(H)-GluN1. The GluD2-LBD and GluD2-LBD-Y770F-(H)-GluN1 are being purified using His-Trap, Hi-Trap and size exclusion chromatography, which will be the focus in the poster.

Determination of phase transitions in Li-ion battery materials using synchrotron operando PXD

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Rechargeable batteries are expected to be a major component of energy storage for a society based on renewable energy. Lithium ion batteries are the most promising battery type in this regard, and the technology has seen extensive research in the last two decades. Li$_3$V$_2$(PO$_4$)$_3$ is a well-known candidate as a cathode material in rechargeable Li-batteries, showing good cyclic stability, high operating potential and high theoretical capacity (197 mAh g$^{-1}$). The material exhibits distinct potential plateaus during Li-extraction for the crystallographic distinct lithium ions, which is typically a sign of a multiphase system where each phase determines the potential. However, the Li-insertion mechanism is known to depend significantly on the potential window used during Li-extraction, which determines the number of Li-ions extracted. Specifically, when extracting all three Li-ions, some of the plateaus disappear on subsequent discharge, which may indicate solid solution behavior. The phase transitions are difficult to examine using ex-situ diffraction experiments, since some of the transitions are subtle, with a change in cell volume of only 0.8%. Operando synchrotron diffraction experiments offer the brilliance and penetrating power to better investigate these structural changes in cathode materials. To investigate the nature of the charge/discharge mechanisms, a series of Li$_3$V$_2$(PO$_4$)$_3$ cathodes were prepared and studied by operando synchrotron radiation powder X-ray diffraction using different potential windows. This allowed us to correlate the features observed in the charge/discharge curves with the phase transitions found from diffraction.
Restructuring in block copolymer thin films: In situ GISAXS investigations during solvent vapor annealing
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Block copolymer (BCP) thin films have been proposed for a number of nanotechnology applications, such as nanolithography and as nanotemplates, nanoporous membranes and sensors. Solvent vapor annealing (SVA) has emerged as a powerful technique for manipulating and controlling the structure of BCP thin films, e.g., by healing defects, by altering the orientation of the microdomains and by changing the morphology. Due to high time resolution and compatibility with SVA environments, grazing-incidence small-angle X-ray scattering (GISAXS) is an indispensable technique for studying the SVA process, providing information of the BCP thin film structure both laterally and along the film normal with a time resolution of ~ 1s (for a recent review see [1]).

Examples of SVA of different diblock copolymer thin films with a single solvent and with mixtures of solvents will be presented. The figure below show to the left, part of a phase diagram with the product of the Flory-Huggins interaction parameter, $\chi$, and the overall length of the diblock, N, along the y-axis and the relative size of the two blocks, f, along the x-axis. A thin film of polystyrene-b-poly(dimethyl siloxane) is treated first with toluene, which is slightly selective towards the polystyrene majority block. The structural evolution follows the red path in the phasediagram (the numbers refer to corresponding GISAXS maps shown on the right). After a while, the fraction of heptane, selective towards the poly(dimethyl siloxane) minority block, is increased in steps, with the resulting structural evolution following the green curve in the phasediagram.

Fluorine-Substituted Metal Hydrides for Thermal Energy Applications

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Since the start of the industrial revolution, our energy requirements have exponentially increased due to the rate of our technological progression. Most of these new energy requirements are mainly met by the consumption of unsustainable resources of fossil fuels and only a small part of it by renewable energies [1]. When most people consider the implementation of renewable energy into society, they generally consider methods of energy production. Fossil fuels are basically an energy store which is released upon burning and as such, we need to consider the storage of renewable energy, and not just production. While battery technologies will undoubtedly play an important role in addressing the energy storage challenge, a crucial realisation is that “thermal energy may be stored with higher specific energy density than for any other known practical form of rechargeable energy storage” [2]. Molten salts are the first generation of thermal energy storage materials used for Concentrating Solar Power (CSP) and, as such have their drawbacks including low heat storage capacity, large volumes, high costs, and an operating temperature that is limited to 565 °C [3].

Metal hydrides are compounds that can reversibly react with gaseous hydrogen and store it in a solid state. The absorption of hydrogen is an exothermic process (releases heat) while the desorption reaction is endothermic (absorbs heat). This property of metal hydrides means they have long been investigated for energy related applications [4] and interest in metal hydrides as thermochemical heat storage media for high temperature CSP has recently been revived with the exploration of low-cost metal hydrides based on Na, Mg, Ca, Fe etc. [5]. In particular, we have focussed on Na and Mg based hydrides for thermal energy storage by exploiting “hydride-fluoride analogues” [6] to produce solid-solution hydride-fluoride systems with both higher thermodynamic stability and reduced cost. We used temperature-dependent in situ Synchrotron X-ray diffraction to explore the change in solid-solution composition during the hydrogen desorption and absorption reactions. The low scattering power of hydrogen means that it has traditionally been difficult to obtain accurate hydrogen occupancies via Rietveld refinement. However, by analysing the temperature-dependent lattice parameters of hydride-fluorides with known composition, changes in the solid-solution composition during hydrogen absorption/desorption could be determined based purely on changes in the lattice parameter.

References
Today, the leading car manufacturers focus on on-board high-pressure hydrogen gas storage ($p = 700$ bar). This technology has the advantage of rapid refuelling ($t < 3$ min) of $\sim 5$ kg H$_2$, which provide long driving range of $\sim 500$ km. The drawback of this fast refuelling is the significant amount of the heat of compression generated inside the hydrogen tank. Therefore the hydrogen gas is pre-cooled to $T = -40$ °C to prior to tank filling in order to keep the walls below the critical temperature during fuelling ($T < T_{max} = 85$ °C) in order to preserve mechanical stability of the storage tank. One approach to increase the overall energy efficiency of this on-board high-pressure hydrogen gas storage is to avoid alternatively diminish the need for such pre-cooling of hydrogen. This might be done by using a thermal energy storage material inside the tank. One option is to utilise latent heat storage. The isothermal nature of this storing process is appropriate for hydrogen tanks. Here we present the results on using the phase change material sodium acetate trihydrate encapsulated in resorcinol formaldehyde carbon xerogels as latent heat storage. In situ synchrotron radiation powder X-ray diffraction experiments confirm a successful melt infiltration of sodium acetate trihydrate into the resorcinol formaldehyde carbon xerogels and demonstrate the samples ability to reversibly melt and solidify. The samples are further analysed by combined thermogavimetric analyses and mass spectroscopy evaluating the mass loss, enthalpy and gas evolution of the samples. Furthermore, additional materials are suggested as possible latent heat storage materials.
One step compositional grading in the PbTe-SnTe system

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First proposed by Ioffe [1], property graded materials have by many authors been suggested to better match material properties with actual temperature profiles in thermoelectric generators. A previous study showed how carefully planned crystal growth can be used to achieve graded samples of the GeSi system by a one step process [2]. To further exploit this technique it needs to be proven for other systems as well. Historically group IV telluride based materials are among the most used thermoelectrics for intermediate temperature ranges, and recent years has seen a renewed interest in them as potential zT>1 materials [3].

PbTe and SnTe form a fully miscible pseudo-binary solid solution in the entire composition range. As the band gap decreases when one component is added to the other, the system is ideal for preparing samples graded in band gap. Samples prepared by directional bulk crystallization at intermediate compositions will have a decreasing concentration of PbTe as crystallization proceeds. As PbTe and SnTe have a high tendency to form inhomogeneously, a rocking furnace is recommendable. This can, however, not be used during Bridgman growth. A study of the prospects of achieving a method for preparing graded samples is presented here.

By preparing samples at several composition ranges across the phase diagram, the relation between composition and parameters relevant to thermoelectric performance has been investigated at a wide range of compositions, and the most promising compositions for grading have been identified.

References:
Spin density measurement of water-bridged Co-dimer using polarized neutrons.

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The nature of magnetic super-exchange through M-O-M bonds for spin-only metal ions is nowadays fairly well understood, but for ions with first order spin-orbit coupling in the ground state, the intuitive understanding is lost, and is still to be fully understood.

In an attempt to systematically investigate the magnetic coupling in such systems we have therefore initiated an experimental study of the spin density distributions in a range of simple water-bridged cobalt dimers which have shown ferro- or antiferromagnetic coupling depending on the nature of the ligand, and with only minor changes to the central M-O-M geometry.

In this work, I present results from studies of the two ferromagnetically coupled, isostructural compounds[1], [Co₂(H₂O)(piv)₄(Hpiv)₂(3-pic)₂] (1) and [Co₂(H₂O)(piv)₄(Hpiv)₂(py)₂] (2). The molecular structure of compound 1 is shown below in Figure 1.

The neutron diffraction data were collected at D3 and D9 at ILL, using newly synthesized large crystals. This provided both flipping ratios[2] from polarized data (D3) and the nuclear structure from un-polarized data (D9) enabling refinement of the spin density distribution using a multipole model description, available in Fullprof.

This analysis will determine if there is spin density present on the bridging water-oxygen, and whether this is of same spin as that on Co (spin delocalization) or opposite spin (spin polarization). The results will hopefully enhance our understanding of the exchange coupling between metal ions with first order spin orbit coupling in the ground state.

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Synthesis and characterization of CuCrO$_2$ delafossite nanocrystals

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Delafossite (ABO$_2$) nanostructures, such as CuCrO$_2$ nanoarticles have shown promising properties in dye-sensitized solar cells (DSSCs). Previous studies have shown that changing the metal on either the A or B site in the structure influences both the conditions required to synthesize delafossite compounds and their tendency of forming nanocrystals or bulk particles$^{1,2,3}$. Here, we present a series of syntheses with varying parameters, illustrating how the conditions must be optimized to obtain the pure delafossite CuCrO$_2$ phase. Structure characterization was done through Powder X-ray Diffraction (PXRD) showing change in particle size with increasing synthesis time and temperature. By varying both temperature and time, the amount of secondary phases (especially CuO) in the final product furthermore varied and it was only possible to synthesize pure delafossite when certain criteria regarding pH, temperature and time were met. Rietveld refinement of the PXRD data showed anisotropic nanoparticles with approximate sizes of 130x130x40Å with the trigonal delafossite unit cell. However, the particles are too small to give a satisfactory fit using conventional Rietveld refinement applying Scherrer analysis, and Rietveld refinement cannot give a satisfying fit. Pair Distribution Function (PDF) analysis of X-ray total scattering will therefore be used to obtain a better understanding of the variation in structure with synthesizes parameters.


Nickel oxide nanoparticles have several potential applications in e.g. electrochemical catalysis\(^1\). The properties are highly dependent on crystallite size, and to increase performance, investigations on the particle formation and size control are important. Here, we study the synthesis of NiO nanoparticles in organic solvents, which facilitates the synthesis of monodisperse nanoparticles with high control of crystallite size between 3 and 6 nm. We study two different reaction pathways, using benzyl-alcohol and tert-butanol as solvents. By controlling synthesis parameters such as temperature, reaction time, and precursors, NiO-nanoparticles in different sizes can be prepared. The nanoparticles are characterized using Powder X-ray Diffraction (PXRD) in order to determine the purity of the synthesis product. The nanostructure is furthermore studied using X-ray Total Scattering with Pair Distribution Function (PDF) analysis, where the local structure can be investigated and the crystallite size can be determined. In situ X-ray total scattering will allow elucidating the formation mechanism in the synthesis.

\(^1\)http://onlinelibrary.wiley.com/doi/10.1002/adfm.201303600/abstract
McXtrace 1.4, Newest features and Developments

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We present the new features included in McXtrace release 1.4. McXtrace is an open source ray-tracing package designed for simulating X-ray experiments. Among the new features:

1. A set of sources including Wiggler, and Undulator.
2. Flat & bent Bragg-monochromator crystal components.
3. Full-size mode of DanMAX at MAX-IV.
4. Full-size model of FXE at XFEL.
5. SAS-view based SAXS-sample components.
6. A particle list output interface file-format (MCPL) to enable interfacing with e.g. SimEX and MCNP6.
7. Polycrystal model We show examples of these new features, and how they may be utilized in beamline design as for DanMAX.

... and more.
Cyclam-based Coordination Polymers for Gas Storage and Catalysis

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Metal-organic coordination polymers consist of metal centers connected by rigid organic linkers. By changing the linkers, these frameworks can be designed to have pores of different sizes and shapes relevant to the desired application. Furthermore, the dimensionality of the compound can be adjusted, producing linear (1-D), layered (2-D) or more complex (3-D) structures. Coordination polymers have shown promising applications for gas storage and catalysis.¹

Herein, we present the synthesis of nickel cyclam (Ni[cyclam]) based coordination polymers and molecular complexes with different organic linkers: Oxalic acid, benzene-1,4-dicarboxylic acid (BDC) and benzene-1,3,5-tricarboxylic acid (BTC). The products are 1-D Ni[cyclam]BDC and 3-D Ni[cyclam]BTC porous structures, respectively, as well as a dinuclear macrocyclic complex of Ni[cyclam]oxalate. The materials were structurally characterized, and the magnetic properties of the porous compounds were investigated. Cyclic voltammetry measurements of Ni[cyclam]oxalate has been undertaken to study the potential of such materials for use in electrocatalysis. Moreover, the gas adsorption properties of these compounds will be investigated.

Structural characterization of the thermostable
Bradyrhizobium japonicum D-sorbitol dehydrogenase

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Abstract Bradyrhizobium japonicum sorbitol dehydrogenase is NADH dependent and active at elevated temperatures. The best substrate is D-glucitol, although L-glucitol is also accepted giving particular potential in industry applications. Crystallization lead to a hexagonal crystal form, with crystals diffracting to 2.9Å resolution. In attempt to phase the data, a molecular replacement solution based upon PDB entry 4NBU (33\% identical in sequence to the target) was found. The solution contained one molecule in the asymmetric unit, but a tetramer similar to the one found in other short chain dehydrogenases, including the search model, could be reconstructed by applying crystallographic symmetry operations. A continuous $\beta$-sheet is formed between two monomers in the tetramer, a feature not generally shared by short chain dehydrogenases, and which may contribute to thermostability. The active site contains electron density consistent with D-glucitol, but there is not clear evidence for NADH being bound.
In situ and ex situ annealing studies of hydrothermally synthesized $\text{SrFe}_{12}\text{O}_{19}$ nanoplatelets.
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Strontium hexaferrite is a chemically stable and cheap magnetic compound, free of rare-earth metals. Previous studies have shown that nanostructuring can improve the magnetic performance of strontium hexaferrite, both as a powder and after pellet compaction, which allows for better bulk magnets for use in for instance electric motors and generators.

Hydrothermally synthesized strontium hexaferrite nanoplatelets are annealed under various conditions and characterized using in situ and ex situ Powder X-Ray Diffraction (PXRD). The Scherrer equation is used to obtain apparent crystallite sizes, which in turn are controlled through annealing conditions. Three different in situ setups have been used, two at an in-house cobalt source diffractometer and one at the PETRA-III synchrotron in Hamburg ($\lambda \approx 0.2 \text{ Å}$). The in situ methods are compared with regard to time and peak resolution as well as Q-range, intensity, and availability.

The final strontium hexaferrite crystallite sizes are determined from Rietveld refinement of ex situ collected PXRD patterns. The magnetic properties of the annealed nanopowders are investigated as a function of crystallite sizes. The results reveal that the crystallites preferably grow along the c-axis and the larger c-axis in turn gives a high coercivity.

Figure 1 - 3D colormap of in-situ diffraction patterns collected during annealing at 750 °C. The acquisition time for each frame were 10 seconds. The measurement were started at room temperature and heating begun at frame 9. The raw data were collected at P02.1 beamline at PETRA III
Materials with the approximate stoichiometry $\text{Cu}_2\text{ZnSnS}_4$ with the crystal structure of the mineral kesterite are currently being investigated as promising materials for thin film solar cell fabrication. Direct imaging, along with chemical analysis, can crucially contribute to assess the quality of the process.

A pulsed layer deposited (PLD) sample of kesterite precursors was previously imaged with a scanning electron microscope (SEM) and electron dispersive spectroscopy (EDS), revealing in particular the presence of surface irregularities. In order to possibly observe finer details and analyze the chemical composition, we acquired fluorescence and scattering maps of such sample at the NanoMAX beam line at MAX-IV, currently in commissioning. This experiment was part of the beam line development.

In our poster we report the outcome of this experiment. We show both fluorescence and scattering maps from features that were highlighted. We present an analysis of the acquired data and a comparison with the previously obtained results from SEM and EDS.

Figure 1: Copper and Zinc Fluorescence and Scattering map for scan #21.
Laccases possess a great biotechnological potential due to their ability to oxidize a wide range of substrates, spanning from small toxic phenols to large biopolymers such as lignocellulose. In particular, the exploitation of laccases to increase yields in biorefinery has received a lot of interest. Like other multicopper oxidases, laccases operate via catalytic Cu-centers and coupling of the oxidative reaction to reductive cleavage of atmospheric oxygen into water. Small phenolic substrates have been shown to bind in a pocket near the T1-Cu site. The size and topology of this pocket, however, vary significantly between subclasses of laccases. Furthermore, a novel binding site was, very recently, reported for a non-phenolic substrate. Thus, despite extensive structural and functional studies, details of the catalytic mechanism and substrate recognition mode(s) in these multifaceted enzymes are still not well understood. With focus on the two major classes of fungal laccases and a systematic series of natural substrates, we use a combination of X-ray crystallography, activity assays, bioinformatics and comparative structural analyses to further elucidate structure-function relationships. As part of this study, we have determined the crystal structure of laccase from *Myceliophthora thermophila* (MtL) to a resolution of 1.5 Å, which provides a detailed view of the architecture of the ligand-free laccase, including an array of ordered water molecules in the substrate binding pocket. Efforts are ongoing to produce a series of MtL:ligand complexes to probe the structural determinants of substrate recognition.

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Interest in CuO has recently resurfaced with the discovery of a multiferroic phase at high temperatures, between 213 K and 230 K [1]. Neutron scattering has revealed that the multiferroicity is accompanied by a change in the magnetic structure from a commensurate antiferromagnet at T < 213 K to an incommensurate helical structure in the multiferroic phase [2]. The dominant magnetic exchange interaction is antiferromagnetic along the (10 -1) direction, indicating that CuO can be viewed as pseudo 1-dimensional chains along this direction. Even so, the weaker inter-chain interactions are vital for a complete understanding of the multiferroicity in CuO, and reliable experimental determination of these interaction are needed. The problem has been addressed in a number of ab initio calculations and experiments. However, these studies disagree on the values, signs and relative importance of the exchange interactions, and despite much effort no clear picture has emerged.

In order to solve this problem we have used neutron time of flight experiments to map out the magnetic scattering in the low temperature antiferromagnetic phase, and supplemented with polarized neutron scattering in both phases. We find the dynamics along the chains to be 1-dimensional, displaying a continuum of scattering in accordance with the des Cloizeaux-Pearson dispersion for an ideal $S = 1/2$ antiferromagnetic chain [3]. We further probe the inter-chain dynamics and have developed a linear spin wave model using SpinW [4] that provides an excellent description of the experimental magnon dispersion, allowing for an accurate determination of the relevant exchange constants. I will present the neutron scattering data together with details of the spin wave model and use this to provide an understanding of the different ordered phases in CuO.

ZnSb is a promising thermoelectric material due to low cost, non-toxicity and low thermal conductivity. The low thermal conductivity in ZnSb, which is comparable to established thermoelectric PbTe, could originate from its chemical bonding. Madsen and coworkers found the Sb-Sb dumbbell rattling model is unlikely because low-energy optical modes involve both Zn and Sb vibrations [1]. Recently the electron-poor bonding properties of ZnSb is argued as the origin of the low thermal conductivity [2]. ZnSb is an electron-poor II-V semiconductor with 3.5 valence electrons per atom, which is less than 4 valence electrons per atom in tetrahedrally bonded semiconductors. Since the charge transfer calculated from theoretical charge density is only ±0.27, chemical bonding in ZnSb is covalent rather than ionic [3,4]. The structure of ZnSb (space group: Pbc a, Z = 8) consists of planar rhomboid Zn2Sb2 rings which are connected to the other rings by Zn-Sb or Sb-Sb. Each atom has a fivefold coordination and topological analysis of the theoretical charge density shows these are bonding interactions [2]. It has been considered that the Zn2Sb2 ring built by 4 center 4 electron (4c4e) bonding is connecting by 2 center 2 electron (2c2e) bonds [4,5]. This bonding picture has been discussed based on the theoretical charge density. It is very important to understand the peculiar chemical bonding in ZnSb, which is related to physical properties. However, the nature of each chemical bond has not been fully discussed.

In the present study, we evaluate the chemical bonding in ZnSb based on experimental charge density. The charge density was obtained by the extended Hansen-Coppens multipole modelling [6] using single-crystal diffraction data at 20 K with a resolution of sinθ/λ < 1.67 Å⁻¹ measured at BL02B1 of SPring-8. In the presentation, we will discuss the electron-poor chemical bonding and connect it to the physical properties.

Multi-scale, in-situ mapping of defect structures in ferroelectrics

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Ferroelectric materials exhibit strong coupling between electric field and strain, making them essential to many medical and electronic applications. Underpinning this are hierarchical networks of structural defects such as grain boundaries, dislocations and domain walls that distort or reorganize in response to electrical and mechanical fields. Measuring such structural dynamics is essential to developing new materials, but remains a persistent challenge. Using in-situ dark-field x-ray microscopy [1] to map lattice distortions around deeply embedded defects, we reveal how these defects create unexpectedly long-range strain fields up to several µm – orders of magnitude more than generally assumed. Imaging these defects in real-time during phase transitions and electric field application then shows how such heterogeneous structural distortions affect material functionality. Such extrinsic strains are pivotal in defining local properties and the self-organization of defects, and must be accounted for by emerging computational approaches to material design.

Figure 1. Dark-field x-ray microscopy maps of ferroelectric defect structures: (a) misorientation in a grain of BaTiO3 [2], (b) strained domain walls in a KNbO3 single crystal [3], (c) individual screw dislocations in a BiFeO3 film [4], and (d) local dielectric polarization tilting around single edge dislocation in the same BiFeO3 film [4].

References
Group 13 metal oxide formation, transformation and pre-nucleation:

A study of the indium oxide system

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Several metals, such as the group 13 and certain transition metals, form different oxide compounds of the forms M(OH)$_3$, MOOH and M$_2$O$_3$, often with multiple phases. To investigate the formation of these compounds the indium oxide system has been examined, to compare with similar results for aluminum and iron from literature.

From Rietveld refinements of samples synthesized by sub- and supercritical flow synthesis it was clear that InOOH formed at lower temperatures and In$_2$O$_3$ at higher, independent of the solvent being H$_2$O or ethanol.

In-situ total scattering data was measured on the system to gain an understanding of the formation of the different phases via PDF analysis. This is an excellent tool to gain information about pre-nucleation clusters and the amorphous and crystalline contributions during formation and phase transformation. The initial pre-nucleation cluster was found to be two corner sharing In-O octahedra, upon heating, nucleation and growth was probed for both InOOH and In$_2$O$_3$.

Synthesis and Characterization of Cyclam-based MOFs for Catalytic Applications

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Metal-organic frameworks (MOFs) are a class of hybrid materials with 1D, 2D or 3D structures. Due to their high surface area, porosity and the possibility of fine chemical tuning, there is an increasing interest in using MOFs for catalysis. The focus of this study is to investigate the catalytic properties of cyclam-based MOFs.Electrocatalysis of cyclam-based materials is a promising, efficient and low-cost method to reduce CO2\(^1\).

In this ongoing project, a cyclam-based MOF\(^2\) with a light absorbing bipyridine dicarboxylate linker with open 2,2-bipyridine sites was synthesized and characterized. The linker interacts with the nickel cyclam motifs to form an open MOF with a rhombohedral crystal structure consisting of linear chains oriented in different directions as seen in the figure. The crystal packing results in a microporous structure. The linker was chemically functionalized by postsynthetic modification to enhance the catalytic activity. The gas adsorption and photocatalytic properties of the materials are investigated.

Figure 2: The overall framework structure consists of three series of linear chains extending in different directions.

Synthesis of various metal borohydrides

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Metal borohydrides have been extensively investigated over the last few years as potential hydrogen storage materials for mobile applications, due to their high gravimetric and volumetric hydrogen content, e.g. 18.5 wt% hydrogen in LiBH₄.¹ Unfortunately the lightweight alkali metal borohydrides have challenges due to their high desorption kinetics and limited reversibility at moderate conditions.²,³,⁴

In this work, we present a new approach to synthesize halide- and solvent free metal borohydrides starting from the respective metal hydride. The synthetic strategy ensures that no metal chloride or LiBH₄ is present in the sample. The synthesis pathway has been shown to work for most of the already known metal borohydrides, M = Na, Ca, Sr, Ba, Y, La, Eu, Gd, Tb, Dy, Ho, Er, Tm, and Yb, but also new borohydrides are formed, M = Pr, Nd and Lu. Besides new compounds, new polymorphs of the rare-earth metal borohydrides are found, all crystallizing in the α- and β-Y(BH₄)₃ structure (except for La(BH₄)₃).

The synthesis pathway start with hydrogenation of the metal. The formed metal hydride is then activated by high energy ball milling to increase reactivity. The next step involves solvent based Schlenk techniques, where the metal hydride and dimethyl sulfide-borane complex (DMS-BH₃) is mixed in appropriate ratios, and left to react at 45 °C, typically for a few days, whereby full conversion to the metal borohydride is generally achieved. For the trivalent M(BH₄)₃, DMS coordinates to the metal. Hence, the powdered M(BH₄)₃-DMS is heated to 140 °C for 4 hours to obtain pure M(BH₄)₃.

The rare-earth metal borohydrides have been investigated by infrared spectroscopy and thermal analysis (TGA-DSC-MS). Furthermore, the structural trends are investigated by synchrotron radiation powder X-ray diffraction. Rehydrogenation properties are investigated for Tb(BH₄)₃ and Lu(BH₄)₃ using in-house PCT equipment. The decomposition pathway of the rare-earth metal borohydrides seems similar, but Lu(BH₄)₃ stands out by decomposing in two steps, in contrast to the one-step decomposition that are observed for the remaining.

References:

Optimization of spring exchange coupled ferrites, studied by in situ neutron diffraction

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Strong permanent magnets with a high energy-product are vital for a great number of electronic devices, these can be found in transformers, loudspeakers, windmills etc. Normally the preferred type of magnets are Rare Earth Metals (REM) containing magnets. REM excels since the magnetic contribution origins from the 4f orbitals which can contain up to 14 electrons. But since REM are both expensive and difficult to mine, a great demand has come for cheaper types of magnets with a similar magnetic performance.

A candidate could be the transition metal oxides. Here the magnetic contribution origins from the 3d orbitals but these can only contain up to 10 electrons. This means that other measures have to be done in order to compete with the REM magnets.

One prominent method is mixing a hard and a soft magnetic phase, on the nanoscale, to achieve an exchange coupling between the phases and enhancing the magnetic energy product. For the exchange coupling to happen it is crucial to have the right ratio between the hard and the soft phase but also to control the size of the particles since exchange coupling is a very small range effect.

In this study, nanoparticles of the spinel CoFe$_2$O$_4$ (hard magnet) is reduced to a metallic alloy CoFe (soft magnet) by heating the sample and flowing it with hydrogen gas. It is studied in situ using neutron powder diffraction with a time resolution of 12 min. The transition from spinel to pure metal goes through an intermediate step of a metal oxide before being fully reduced. These metal oxides are antiferromagnetically ordered an is therefore considered a parasitic phase. However by fine-tuning the reaction temperature and hydrogen flow rate the occurrence of the phase can be minimized.

In order to distinguish between Co and Fe Neutrons are chosen. Since neutrons have a spin it will also be possible to measure a magnetic signal and investigate the exchange-coupling. After the reduction the samples was furthermore investigated using powder x-ray diffraction and VSM (vibrating sample magnetometer).

To understand the reaction mechanism, a series of experiments with varying temperature (fixed flow) or varying flow (fixed temperature) has been performed.

To optimize the exchange-coupling several experiments with fixed temperature and flow, have been performed where the conversion from spinel to metal has been varied.
Pair distribution function analysis of Jahn-Teller fluctuation in copper Tutton salt

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The Tutton salt is a group of inorganic salts with a general formula of $A_2[M(H_2O)_6]XO_4$. $A$ is an alkaline ion or ammonia, $M$ is a transition metal from the 4th period and $X$ is either S or Se. The copper Tutton salt contains a Jahn-Teller distorted octahedral and ammonia and sulfate as the counterions.\[^{[1]}\] At low temperature only one of the three axes in the octahedral is Jahn-Teller distorted though as the temperature is increased the distortion fluctuates between two axes in the octahedral.

In this study the time and space averaged structure of the powder X-ray diffraction pattern (PXRD) is compared to the time averaged structure obtained from pair distribution function (PDF). The different regions of the PDF (low and high r regions) are investigated to gain insight to the accuracy of which the PDF can predict the average and local structure.

In the figure are the octahedral $\text{Cu(H}_2\text{O)}_6$ and the tetrahedral $\text{SO}_4$ and $\text{NH}_4$ shown. The anisotropic atomic displacement parameters are investigated for rocking modes induced by the fluctuation of the Jahn-Teller distortion.

Cu (blue), O (red), S (yellow) and H (white). The position and isotropic atomic displacement parameters of H have been determined by Maslen et al.\[^{[2]}\]

References:


X-Ray tomography studies on all-solid-state Li-S batteries built around LiBH4 solid electrolyte

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Efficient energy conversion and storage is crucial for development of systems based on renewable energy sources. For electricity storage, Li-ion batteries are commonly used in electronics devices but require many improvements to obtain longer life-time and higher energy densities. The current use of organic liquids and gels electrolytes limits these improvements because of lithium dendrites formation, reducing the lifetime of the battery and which can possibly be hazardous due to risks of short circuits.

Many alternatives to current state-of-art lithium-ion batteries exist; among them are lithium-sulfur solid-state batteries, solid electrolytes having higher stability when compare to liquid electrolytes, with no risks of vaporization and leakage while sulfur cathodes have large theoretical energy density. LiBH4 is a promising material for solid-state batteries as it is lightweight and stable electrochemically at least up to 6 V. While the orthorhombic phase (Pnma), stable at room temperature has a low ionic conductivity (~10⁻⁵ mS cm⁻¹ at 30 °C), the hexagonal phase (P63/mmc), stable above 110 °C, has a much higher ionic conductivity (~1 mS cm⁻¹ at 120°) [1]. Confinement of LiBH4 in mesoporous SiO2 allows obtaining fast ionic conductivity even at room temperature.

We have successfully built and cycled solid-state lithium-sulphur batteries based on LiBH4 and have performed in operando X-ray diffraction, -tomography and Raman spectroscopy measurements on capillary cells of 1 mm in diameter. Figure 1 shows a capillary cell with an image obtained from X-Ray tomography. Figure 2 shows reconstruction of a Li-S capillary cell from cross-sectional images. X-Ray and Raman data deliver information on the electrochemistry of the battery helping to understand and improve the battery performances.

Figure 1. Li-S capillary cell – X-Ray tomography

Figure 2. Reconstruction of a Li-S capillary cell from cross-sectional images

The interest in new battery materials, which are able to “fill the shoes” of the superior lithium ion batteries (LIBs), has increased the recent years. Properties capable of withstanding the increased demand for faster, better and cheaper solutions must be fulfilled in the new battery chemistry. As a suitable candidate for the replacement of LIB employing graphite negative electrodes, sodium-ion batteries (NIB) have long been investigated due to their intrinsic similarity to LIB, e.g. comparable electrode potentials to that of LIBs. However, due to unfavorable thermodynamics, sodium ions have been found unable to intercalate into graphite unless electrolyte solvents are co-intercalated. Fortunately, hard carbons, or non-graphitic carbons, have been successfully used as anodes. The hard carbons consist of few layers of stacked graphene layers, which due to the non-periodicity results in an amorphous structure. The intercalation process upon discharge has through combination of operando $^{23}$Na solid-state NMR and ex situ PDF analysis been characterized to involve a three-stage mechanism. This mechanism is characterized by absorption of sodium ions onto pore surfaces, at defect sites and between expanded layers of graphene, which is illustrated in Fig. 1. However, the previous studies have only been exploring the mechanism through a few ex situ samples and therefore the exact mechanism is still not completely known. Therefore, questions concerning the mechanism arise; how can the nature of hard carbon upon sodium intercalation be characterized? Can similarities to the well-known lithium-graphite system as visualized in Fig. 2 be drawn, when investigating the local structure of hard carbon upon sodiation and de-sodiation? Eventually, can the difference in intercalation mechanism between lithium and sodium be deduced? Based on these questions, the aim of this study has been developed, where detailed structural and microstructural information ex situ and in situ about the intercalation process of sodium ions in hard carbon is intended. As a consequence of the amorphous state of the material and the presence of low Z elements, detailed analysis using total scattering data is used for PDF analysis in order to obtain information regarding the local structural changes during at different stages of sodium intercalation and upon battery operation.
Mapping grain morphology and orientation by Laboratory Diffraction Contrast Tomography

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Recent developments of the Laboratory Diffraction Contrast Tomography (LabDCT) have extended its capabilities to include full reconstruction of the 3D grain structure, including both grain morphology and crystallographic orientation, in the bulk of a polycrystalline sample.

LabDCT makes use of high-resolution diffraction images acquired on a ZEISS Xradia 520 Versa X-ray microscope. The diffraction signals are based on polychromatic X-rays and acquired in a special Laue-focusing geometry that helps increase the signal-to-noise ratio. The unique non-destructive 3D crystallographic imaging capabilities of LabDCT complements the structural data obtained by traditional absorption-based tomography and together they provide an unprecedented insight into materials structure.

We will present a selection of results of LabDCT with particularly emphasis on its non-destructive operation, demonstrated through 4D evolutionary studies obtained by repeating the imaging procedure numerous times on the same sample. We will discuss the boundary conditions of the current implementation, compare with conventional synchrotron approaches, point to the future of the technique and discuss ways in which this can be correlative coupled with related techniques for a better understanding of materials structure evolution in 3D.

Figure 1: (a) 3D reconstruction of an AlCu polycrystalline sample. Grains colors are function of their orientation (IPF coloring). (b) Visualization of a cluster of 6 grains embedded in the bulk of the sample (same color code)
Cu₂ZnSnS₄ (CZTS) thin film solar cells are cheap, non-toxic and present an efficiency up to 9.2% [1]. They can be easily manufactured by the deposition of the nanoparticle ink as a thin film followed by a thermal treatment to obtain large grains [2]. Therefore, CZTS has the potential to revolutionize the solar energy market.

However, to commercialize CZTS nanoparticle thin films, the efficiency issues must yet be resolved. In order to do so, it is vital to understand in detail their nanoscale atomic structure. CZTS crystallize in the kesterite structure, where Cu and Zn is distributed between the cation sites in the structure. The cation distribution affects the properties of the CZTS nanoparticles.

Here, we use the hot-injection synthesis method to prepare CZTS nanoparticles of different compositions. Information on the atomic structure is obtained by combining Rietveld refinement of Powder X-ray Diffraction data with X-ray total scattering with Pair Distribution Function analysis. Powder neutron diffraction will furthermore allow characterization of the cation disorder on the metal sites in the kesterite structure. The nanoparticle ink is also characterized by XRD, EDS, and Raman spectroscopy in order to fully detect possible secondary phases and characterize the CZTS phase.


Upgrade of the Primary spectrometer for RITA-II, PSI

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RITA II is a former RISOE multiplexed neutron spectrometer, currently located at the Paul Sherrer Institute, Switzerland with a continued strong connection to the Danish scattering community. In 2018 the analyzer-detector module will be upgraded to a new CAMEA backend, improving the coverage a factor 150 and the Energy resolution a factor 2. This will leave the primary spectrometer with an unmatched energy resolution and an obsolete guide. To rectify this the guide will be upgraded to a new ballistic guide with a virtual source and the monochromator replaced with a double focusing monochromator. This upgrade will provide a factor 6 performance increase that can be distributed between flux and resolution depending on experimental considerations, while the virtual source will reduce the background considerably. After the full upgrade in 2019 Danish users will enjoy privileged access to a powerful new instrument with up to 2000 times higher performance than the current instrument and providing valuable insight to the BIFROST, instrument constructed by in Denmark for ESS.
Anisotropic magnetoresistivity from van der Pauw measurements on LaSb$_2$

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The Fermi surface of a compound is important in many branches of materials science, and measuring the anisotropic magnetoresistivity provides an experimental path to the Fermi surface$^1$. Deploying the recently developed method for extracting the resistivity tensor from van der Pauw (vdP) measurements$^{2–4}$ allow quick access to the even part of the magnetoresistivity tensor. This poster presents a proof of concept measurement of the anisotropic magnetoresistivity by use of this method with LaSb$_2$ as a model compound. The magnetoresistivity of LaSb$_2$ is by itself interesting as it is linear which is interesting for magnetic sensor applications$^5$. Resolving the directional dependence of this magnetoresistivity could prove useful for such application, while also shedding some light on the origin of the linear magnetoresistivity.

Figure 3: On the left the directionally resolved magnetoresistivity of LaSb$_2$ in the a,b plane with the magnetic field parallel to the c-axis. On the right the directionally resolved resistivity in the a,b plane as a function of temperature.


Core Electron Deformation in Silicon from Powder X-ray Diffraction

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Traditionally Single Crystal X-Ray Diffraction (SCXRD) is used to obtain structure factor amplitudes for Electron Density (ED) modelling in crystalline materials. However, for SCXRD there are some pertinent issues with systematic errors such as extinction, absorption and scaling between detector frames. All three issues can be at least partially solved by performing Powder X-Ray Diffraction (PXRD). Two major issues are, however, present in PXRD; peak overlap and background treatment.

Recently, we have shown that PXRD can be a valuable alternative to SCXRD for ED determination in high symmetry inorganic materials, and the subtle deformations in core ED upon bond formation have been determined for diamond, silicon and cubic boron nitride. For silicon and diamond, the extracted structure factors were shown to be of comparable precision and accuracy to the dynamical Pendellösung data [1,2]. This was made possible by performing diffraction in vacuum with the first version of our custom built Aarhus Vacuum Imaging plate Diffractometer with a sample-to-detector distance of 300 mm. We have now finished commissioning of a new version of AVID with sample-to-detector distance increased to 1200 mm, which is shown to give an improvement in signal-to-background ratio and instrumental peak broadening by a factor of three due to the differences in radial dependences of coherent and incoherent scattering [3].

Benchmark PXRD data on silicon has been collected on the new AVID showing Bragg peaks to an unprecedented resolution of $\sin \theta/\lambda \sim 2.4 \text{Å}^{-1}$ at 100 K. Data are modelled using a combined HC-Rietveld method, where overlapping reflections are partitioned based on the Hansen-Coppens multipole model, which reduces bias towards the independent atom model. The extracted structure factors are compared with the previous AVID and shown to be of significantly improved precision and accuracy at high angles. These are used for modelling the ED in silicon including deformations in the core ED with improved accuracy compared to previous studies.

References:
A NaAlH$_4$-Ca(BH$_4$)$_2$ Composite System for Hydrogen Storage

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Hydrogen as an energy carrier has been considered for decades now owing to its unique properties e.g. the high gravimetric energy density of ~120 kJ/g (lower heating value) [1-3]. A combination of the two well studied compounds, NaAlH$_4$ and Ca(BH$_4$)$_2$, is the focus of the present investigation. The reactive hydride composite, NaAlH$_4$-Ca(BH$_4$)$_2$, contains 9.77 wt% of hydrogen and is thus worth attention.

Mechanochemical treatment (ball-milling) of NaAlH$_4$-Ca(BH$_4$)$_2$ mixtures leads to partial formation of NaBH$_4$ and Ca(AlH$_4$)$_2$ by a metathesis reaction:

$$2\text{NaAlH}_4 + \text{Ca(BH}_4)_2 \rightarrow 2\text{NaBH}_4 + \text{Ca(AlH}_4)_2$$

The reaction proceeds to different extents depending on the applied ball-milling times, which is confirmed by powder X-ray diffraction and infrared spectroscopy, see Figure 1. Additionally, an in-situ synchrotron radiation powder X-ray diffraction (SR-PXD) study reveals that the reaction continues due to thermal treatment.

Finally, the reactive hydride composite system was investigated by mass spectrometry and Sieverts’ measurement, which reveal release of ~6 wt% H$_2$ at $T < 375$ °C.

![Figure 1. FT-IR spectra of NaAlH$_4$-Ca(BH$_4$)$_2$ ball-milled at different ball-milling times.](image)

Core-shell TiO$_2$@MoS$_2$ for Li-ion Battery Anodes

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In the search for novel anode materials for Li-ion batteries with improved performance in terms of capacity, cyclability and environmental friendliness, the TiO$_2$@MoS$_2$ composite has in recent years received much attention.\textsuperscript{[1-4]} An increasing number of experimental studies show that TiO$_2$@MoS$_2$ composites of various morphologies maintain the high lithium storage capability of the layered MoS$_2$ without the loss of capacity upon cycling that often accompanies pure MoS$_2$.

While the capacity and cyclability of TiO$_2$@MoS$_2$ are excellent, the reported synthesis routes have so far been rather labor intensive requiring multiple steps, of great inconvenience for commercialization. In this study, TiO$_2$@MoS$_2$ for Li-ion batteries has been synthesized in a dual stage continuous flow reactor and in a flow reactor with injector pump, offering a sustainable and highly scalable synthesis route. If a composite material with similar excellent electrochemical performance can be produced through this strategy, a large step closer to commercialization has been taken.

Transmission electron microscopy image of TiO$_2$@MoS$_2$ synthesized in a flow reactor with injector pump. The layered MoS$_2$ are surrounding the TiO$_2$ nanoparticles in a core-shell manner.

Understanding protein-protein interactions relevant for neuronal spreading of alpha-synuclein

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Parkinson’s Disease is characterized by dopaminergic neurodegeneration and the presence of Lewy Bodies, whose major constituent is fibrillated α-synuclein (aSN). The native function of aSN is not well-understood, but aSN is believed to be implicated in synaptic exocytosis via SNARE protein complexes and as a membrane curvature-sensor. aSN is an intrinsically disordered protein and native aSN aggregates into the amyloid fibril structures observed in Lewy Bodies via complex structural changes and aggregation pathways. The spreading of amyloid fibrils and/or pre-fibrillar forms of aSN between neurons causes the gradual degradation of particular areas in the brain, starting from substantia nigra and slowly infecting larger parts.

Recently, it has been shown that pre-formed fibrils of aSN can spread to neighboring neuronal cells via interaction with the lymphocyte-activation gene 3 (LAG-3), resulting in endocytotic events (Mao, Ou et al. 2016). We are pursuing a structural understanding of the relevant interactions between LAG-3 and aSN, and the current status of the project will be presented.

The Danish Industrial Neutron Laboratory

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Currently, international state-of-the-art large scale facilities are being built close to Denmark with considerable Danish investments: MAX IV and ESS in Lund and the European XFEL in Hamburg. These new research facilities provide unique possibilities for the characterization and development of technologies and products within materials science, nanotechnology, food science and pharmaceutical science.

The Danish Industrial Neutron Laboratory has been launched to build up a long-term sustainable industrial user service platform at the Danish GTS institutes (Godkendt Teknologisk Service – Approved Technological Service). The aim is to ensure that industry benefits as much as possible from these investments and adopt analysis from large-scale facilities as an integral part of their research and development. The initiative is based at the Danish Technological Institute and collaborates with the LINX project.

Many companies are not aware of the opportunities and potential benefits of carrying out experiments and analysis with neutrons or X-rays. Therefore, the Danish Industrial Neutron Laboratory offers support and guidance for potential industrial users based on their needs through a strong team of specialists. The technical areas have been selected by cross-matching Danish research strongholds as defined by the Danish ESS Lighthouses with industrially relevant technological areas. The following five focus areas have been selected:

- Residual stresses in metals
- Industrial coatings
- Polymers
- Functional nanoparticles
- Bio-inspired materials

Planned actions include:

- Spreading knowledge about potential industrial usage of neutron and X-ray facilities.
- Supporting Danish companies in the relevant stages of using advanced characterization methods, from the assessment of applicability and feasibility, through execution of experiments, to the analysis and interpretation of results.
- Developing a business model for the offer of large-scale facility characterization as an industrial service.
Unliganded and substrate bound structures of the cellooligosaccharide active lytic polysaccharide monooxygenase LsAA9A at low pH

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Lytic polysaccharide monooxygenases (LPMOs) are a new class of copper metalloenzymes that degrade polysaccharides oxidatively and act in synergy with hydrolases to break down biomass (1,2). Exploiting the fact that some LPMOs are found that can efficiently degrade oligosaccharides as well as polysaccharides major progress has also been made in characterizing in details substrate interactions by crystallography (3) and NMR spectroscopy (4). In particular the crystallographic studies of the LPMO from *Lentinus similis* belonging to the fungal LPMO family AA9 have provided the first atomic resolution view of substrate-LPMO interactions and together with complementary biochemical and spectroscopy studies given us a much better picture of the initial stages of the mechanism.

The initially published structures of *Ls*AA9A are determined at pHs close to the ones used for activity studies, however in the course of the structure determination process, we have also determined the high resolution structures of this enzyme at pH 3.5, both in the absence and presence of cellohexaose (5). There are interesting differences between the structures determined at different pHs. For the low pH structure without substrate bound, disorder of the second His in the histidine brace is observed. Differences between cellohexaose complexes determined at two pHs are also visible, although both His of the histidine brace are ordered in this structure. While the full implications of the structures can be understood only when complementary biochemical data become available, these structures give some insights in how LPMO structure and function can be profoundly affected by pH.


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SPAnPS – The Radiant Polymorphs

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In 2003, Fei et al. [1] published an article on the fluorescence properties of trans-9,10-bis(diphenylthiophosphoryle)-anthracene, short: trans-SPAnPS@toluene. Solid-state fluorescence was found whose origin seemed to be exclusively related to the intercalation or co-crystallisation of toluene. In solution, the compound was non-emissive. Moreover, the loss of toluene from the crystal framework upon vacuum drying was directly indicated by the vanishing of the fluorescence. The re-exposure to toluene readily restored the emission and on this basis an exciplex (excited complex) formation was suggested. A detailed charge density investigation is presented that was performed on the trans-SPAnPS@toluene (P21/n) co-crystal in order to further investigate the role of the intermolecular host/guest interactions. Additionally, a variety of orthogonal strategies is combined in order to shed light on the complex mechanisms that take place in the solid form of trans-SPAnPS@toluene [2]. The investigation included single crystal as well as powder X-ray diffraction, neutron diffraction, solid-state NMR and computational chemistry using different levels of theory. Hereby, new and complementary aspects were added to the discussion and, until now, more than 20 polymorphs and co-crystals were discovered. The solid-state NMR chemical shifts supported by the GIPAW DFT method [3] and topological features from the experimental charge density were used to draw a more complete picture of the forces acting in the solid-state forms of SPAnPS. Finally, a rationale for the solid-state fluorescence behaviour of the investigated compounds is suggested.

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References:

Protein drugs are increasingly important in drug development worldwide due to their high specificity, potency, and low toxicity. However, many protein drugs suffer from inherent physical instability limiting the manufacturability and formulation development,[1] as well as a short plasma half-life that is incompatible with delivery of an efficacious dose within the appropriate dose regime. Association to Human Serum Albumin (HSA) is a strategy that has been shown to increase the plasma half-life of protein drugs.[2] Likewise, association to recombinant HSA (rHSA) is a strategy to stabilize protein drugs during manufacturing and in formulation leading to a longer drug shelf life.[1] Insulin detemir (detemir) is an example of an acetylated insulin analogue that associates to HSA.[2]

In this work, complex formation between rHSA and detemir and how it is influenced by physicochemical properties of different formulations have been investigated using an interdisciplinary approach that combines light scattering, small-angle X-ray scattering (SAXS) and molecular dynamics (MD) simulations.

From SAXS experiments, complex formation is found to depend on fatty acid content and ionic strength of the buffer, as well as protein concentration. From SAXS rigid body modelling, a model structure of the complex has been suggested and its stability and protein-protein interactions have been evaluated by MD simulations. Furthermore, dynamic light scattering experiments have been carried out to support the findings in the SAXS experiments.

Acknowledgments

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References

Tailoring metal-organic frameworks: Connecting Co$_4$O$_4$ cubane complexes with the right organic linker

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Splitting water into H$_2$ and O$_2$ by visible light photocatalysis is of major interest in the search for clean energy technologies.\(^1\) In Nature, the CaMn$_3$O$_4$ cubane-like complex plays a key role in photosystem II as catalyst for the water oxidation reaction\(^2\) i.e. the oxygen evolving half reaction of water splitting. Therefore, this system has served as inspiration for numerous studies of multinuclear transition metal cubane-like complexes.\(^3,4\)

A controlled way to synthesize molecular Co(II) based cubanes with a bis-benzimidazole derivative of S-malic acid as ligand has been reported by Deville \textit{et al}.\(^5\) We propose to integrate these cubane units into a metal-organic framework (MOF). Thereby, we investigate how the properties of the cubane core are affected by the organic linker used to connect the Co$_4$O$_4$ units into a metal-organic polymer.

Fundamental questions on the structural topology with a varying organic linker arise: Can we control the assembly of 1D, 2D, or 3D networks? As of now, we have succeeded to produce polymeric chains of cubanes with multiple bifunctional organic linkers by solvothermal synthesis. The next step will be to test their photocatalytic abilities in the water oxidation reaction.

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Hydrothermal liquefaction in a continuous-flow reactor – what could possibly go wrong?
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With crude oil being a finite resource whose consumption is expected to increase in the future, bio-oils made from biomass are an interesting economical as well as ecological alternative. [1] Hydrothermal liquefaction (HTL) utilizes the unique properties of subcritical water to convert biomass into comparatively high-quality bio-oil.[2]

Most studies on HTL are conducted on batch reactor systems, which provide fast and easily conducted experiments. However, the small reactor volumes reduce the analytical possibilities of the products and make these reactors unsuitable for industrial use.

Aarhus University has a custom-built continuous-flow reactor that processes up to 1.2 L biomass slurry per hour. Its construction has been described in detail by Mørup et al.[3] The operation of the reactor, however, has its own challenges which are described in this poster. Starting at the biomass slurry preparation, addressing possible locations for clogs and discussing sources of experimental errors, these challenges are by no means limited to this specific reactor and can help to improve future continuous-flow reactors.

Mesoporous silica as an oral adjuvant for hepatitis B vaccination

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Mesopores silica SBA-15 is a promising candidate for a new adjuvant for oral delivered vaccines, such as Hepatitis B, diphtheria, tetanus and more. Other than an improved immunological response and lower toxicity [1], encapsulation in SBA-15 has other practical benefits such as easier storage and easier administration when compared to standard vaccine delivery methods, i.e. by injection.

Hepatitis B surface antigen used in vaccination against hepatitis B is a curious case as the antigen is too large to enter the mesopores of SBA-15, but is still protected from the gastric acid. Thus inducing good immunological response when administered using the SBA-15 as adjuvant[2].

In this work we have used X-ray and neutron tomography to shed light in how the hepatitis B antigen is being protected in the macroporosity, with sizes larger than 50 nm, that is created by the rod shaped SBA-15 particles. Tomography results were also used to determine the optimal ratio of antigen to SBA-15 in order to prevent agglomeration of the antigen.

By means of small angle X-ray scattering (SAXS) we were able to demonstrate that the protected antigen in the macroporosities is attaching on to the opening of the hexagonal ordered mesopores. Finally, in-situ SAXS experiments show that when soaked onto the mesopores the antigen is protected from the gastric acid and is released only when entering the intestinal environment.

References:


X-ray nano tomography of Hepatitis Antigen marked red, encapsulated in the macroporosities of SBA-15 silica.
Small-angle scattering study on the structure of lecithin-stabilized tetracosane-water nanoemulsions and suspensions

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The structure of the interfacial stabilizer layer in lipid emulsions and suspensions is relevant i.a. to the lipid oxidation of encapsulated bioactive compounds and to the crystallization of the nanoemulsions. In this study tetracosane (C$_{24}$ alkane, TCS) model nanodispersions stabilized by the lecithin 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC) were considered. The TCS o/w nanoemulsions were prepared by high-pressure melt homogenization. The droplets (about 65 nm in diameter, as measured by photon correlation spectroscopy) exhibit a strong super-cooling (ΔT about 20 K) and crystallize in an for TCS unusual orthorhombic crystal structure (space group Pca2$_1$ as verified by wide-angle x-ray scattering). Using small-angle x-ray and neutron scattering and nanodispersions with different neutron scattering contrasts for the TCS core and the DMPC stabilizer layer, the molecular arrangement of DMPC in the interfacial layer was studied. For the nanoemulsions a dense monolayer of DMPC with a thickness of about 16 Å was found with only a minor interpenetration between TCS and the acyl chains of DMPC. For the nanosuspensions a monolayer thickness of about 10 Å is found, indicating a more flat arrangement of the DMPC molecules at the interface. This could be explained by the expanded surface of the nanocrystals with respect to the emulsion droplets.

Structure and electrochemical performance of LiVPO$_4$F as a cathode material

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LiVPO$_4$F is a new promising high potential cathode material for Li-ion batteries due to its high capacity, high operating potential and excellent thermal stability. We have synthesized the material by two different synthesis routes: solid state diffusion and hydrothermal synthesis. Electrochemical tests have revealed that the potentials observed during charge and discharge of batteries employing the two cathode materials differ significantly (see Figure 1).

Figure 1. Electrochemical behavior of LiVPO$_4$F prepared by solid state synthesis (left) and hydrothermal synthesis (right). In both figures the voltage in millivolt is shown as the function of time in hours during battery charge and discharge at a current rate of C/5 (corresponding to complete charge or discharge in five hours).

Here we investigate the origin of the difference in electrochemical behavior by studying the material morphology at the nanoscale and in particular the change in atomic structure during the electrochemically driven phase transitions taking place as the battery charges and discharges. This is achieved through operando synchrotron radiation powder X-ray diffraction (at BM01A, SNBL, ESRF) utilizing battery test cells specially designed for operando X-ray scattering studies. Rietveld refinement provides information about the phase transition mechanism as well as changes to the crystalline structures, which are linked to the electrochemical behavior simultaneously monitored during the operando experiment.

Our data reveals that during the charge the triclinic LiVPO$_4$F transforms to a monoclinic VPO$_4$F structure. The transition is reversible during discharge and followed by yet another triclinic → monoclinic transition as LiVPO$_4$F transforms to Li$_2$VPO$_4$F. The difference in electrochemical behavior between the two samples can be linked to differences in two-phase and solid solution behavior of the phase transformation.
Molybdenum oxide nanoparticles have potential applications in catalysis, batteries, gas sensor and in memory materials [1, 2]. In this study, we show how molybdenum oxide nanoparticles can be prepared from a simple solvothermal synthesis. The MoO$_2$ nanoparticles were synthesized from an aqueous solution of ammonium molybdate tetrahydrate, polyethylene glycol and HNO$_3$. Powder X-ray diffraction with Rietveld refinement were used to characterize the product and the structure. Here, we show that the different parameters (synthesis time, temperature, acid and PEG concentration) have a large influence on the product. The particle size can be controlled from 22.50 to 25.98 nm by adjusting the crystallite size as well as the PEG concentration in the synthesis. We furthermore show that the synthesis is only successful if both acid and PEG is present in the precursor. Total scattering with Pair Distribution Function analysis is used to further study this effect.


Modification of light absorption properties of 2D thiostannates

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Photocatalysis is the process of utilizing light energy as driving force of chemical reactions through excitation of a catalyst. Importantly, reactions such as reduction of CO$_2$\(^1\) and H$_2$ production from water\(^2\) can be photocatalysed. By utilizing light energy from the Sun, photocatalysis provides a clean measure to address a number of environmental challenges. As such, a photocatalyst with a broad visible light absorption profile is desirable to best make use of the solar spectrum.

Heterogeneous photocatalysts are typically semiconductors, for which the band gap is the defining factor for the light absorption profile. In this study\(^3\), we have modified the light absorbing properties of the layered, large band gap semiconductor Sn$_3$S$_7$(trenH)$_2$ (tren = tris[2-aminoethyl]amine) by partly exchanging the tren ions with organic dyes in solution, cf. Figure 1, while maintaining the thiostannate structure. To optimize the ion exchange efficiency, a number of experimental conditions has been varied, including temperature, solvent, and dye. The ion exchange follows pseudo-second order kinetics and a Langmuir adsorption isotherm.

So far, the introduction of organic dyes in the thiostannate host has been successful in increasing the visible light absorption, but one question still remains: Will intercalation of dyes improve the photocatalytic efficiency of the thiostannate-based nanocomposites?

Halogenated sodium-closo-dodecaboranes as solid-state ion conductors

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Metal closo-boranes as solid-state electrolytes

Metal closo-boranes [1,2], as well as halogenated derivatives, have shown great potential as electrolytes both in the solid state and in liquids [3-4]. The high solid-state ion conductivity often found in metal closo-boranes is facilitated by the fast reorientational dynamics of the large anion allowing for higher mobility of the cations, thus the ion conductivity increases by orders of magnitude upon phase transition into the highly disordered high temperature phase.

Ion conductivity in Na2B12X12 (X= Cl, Br, I)

Differential scanning calorimetry was utilized to determine the transition temperatures between the room temperature and high temperature polymorph, showing an increasing transition temperature with anion size and mass ranging from 475 °C for Na2B12Cl12 to 570 °C for Na2B12I12. The phase transition was found to be reversible upon cooling with hysteresis of about 50 °C.

Above the transition temperature the compounds showed remarkable ion conductivities, values of 0.014 and 0.162 S/cm for Na2B12Cl12 and Na2B12I12, respectively, are here reported [5].

Crystal structure

The structures of Na2B12Cl12, Na2B12Br12 and Na2B12I12 were determined based on powder X-ray diffraction from both synchrotron and in house sources. All three compounds were found to have a cubic room temperature (α−) and a cubic high temperature polymorph (β−) and to be isostructural with one another. The room temperature structures were described in the space group Pa-3, with a = 12.6496(4) Å, a = 13.1412(7) Å and a = 14.0229(5) Å, respectively, showing an increasing unit cell volume with increasing anion size, as expected. Upon heating to the respective transition temperature a polymorphic transition to the high symmetry space group Fm-3m occurred owing to the highly disordered anions and cations.

Figure 1: Crystal structure of the high temperature polymorph of Na2B12X12 (X = Cl, Br or I)

References
Effect of precursor choice and sintering conditions for W hexaferrite properties

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Permanent magnets are present everywhere in modern life and essential for our way of living. The highest performance magnets are made with rare-earth metals, but with the increasing price and scarcity of these metals, efforts have been made in synthesizing high performance rare-earth free magnets. Hexaferrites are of great interest because of high stability, high anisotropy and favorable cost/performance ratio.

This work focuses on the W-phase hexaferrite which excels with its high anisotropy constant, Curie temperature, and a higher saturation magnetization compared to other hexaferrites. This is believed to be caused not just by super-exchange coupling across one oxygen (Fe-O-Fe) but super-super-exchange across two oxygens (Fe-O-O-Fe). The unit cell is rather complex with dimensions of \( a=b=5.91 \text{ Å} \) and \( c=32.75 \text{ Å} \).

In this work, \( \text{SrZn}_2\text{Fe}_{16}\text{O}_{27} \) was synthesized by a sol-gel autocombustion synthesis method using metal nitrates, citric acid and concentrated ammonia to prepare the precursor gel. The gel was sintered at four different sintering temperatures (1000, 1100, 1200, and 1300 °C) for two hours in a conventional oven. Almost phase pure \( \text{SrZn}_2\text{Fe}_{16}\text{O}_{27} \) was formed at temperatures above 1200 °C resulting in a significant increase of the saturation magnetization from 47 Am\(^2\)/kg to 72 Am\(^2\)/kg. The sample heated at 1000 °C has a coercivity of 292 kA/m, which was decreased to 6 kA/m when the \( \text{SrZn}_2\text{Fe}_{16}\text{O}_{27} \) was formed.

The coercivity is strongly dependent on particle size and it is reasonable that the powder sintered for 2 h at 1200 and 1300 °C have become multidomain particles due to particle growth resulting in a very limited coercivity.
GeTe: focus on the high-temperature phase transition through high resolution XRPD

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GeTe is extensively studied as a phase change material for data storage and for its thermoelectric properties.[1,2] According to previous neutron diffraction studies[3,4] the high temperature phase transition \(R3m-Fm\bar{3}m\) is second-order displacive with an anomalous volume contraction of the unit cell at \(T_c \sim 700\) K. The displacive character of the phase transition has been questioned by recent extended X-ray absorption fine structure, pair distribution function and time-domain terahertz spectroscopy investigations[5-7] which suggest that the phase transition is of an order-disorder type.

In the present study, we show by means of high resolution X-ray powder diffraction data (XRPD) collected a BL44B2,[8] SPring-8, that the phase transition is of a discontinuous and first-order type. The origin of the volume contraction observed at \(T_c\) is also discussed.

Temperature dependent intrinsic defects in tungsten oxide nanorods

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Tungsten oxide nanostructures have a wide range of applications and can be synthesized in numerous shapes, sizes and phases. It is well known that the structure of a material governs the properties of the material, and that defects in the structure can critically change the material properties. Especially tungsten oxides are known to form phases known as tungsten bronzes, where point defects, in form of intercalated atoms or small molecules, drastically alters the properties depending on the defect concentration[1, 2]. The tungsten bronzes are extremely resistant to most acids and is considered as possible catalysts in highly acidic conditions[3, 4]. We here present a simple synthesis for synthesizing approximately 20 nm long WOx nanorods with varying degree of disorder in the crystal structure. While the size of the particles seems unaltered by changing the temperature, the defect concentration is influenced by the reaction temperature and reaches a minimum point when the reaction is done at 300 °C. Using Total Scattering and Pair Distribution methods we perform a structural refinement of the nanoparticle and estimate the degree and type of disorder. The link between synthesis and defects are likely to be relevant for other metal oxide structures and their potential uses.

Figure 1: A Pair Distribution Function of nanoparticles formed under 5 different reaction temperatures. The peak related to disorder in a WO phase is decreasing with temperature to stabilize at higher temperatures.

Study of layered double hydroxides intercalated with para-aminosalicylate

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LDH’s are a group of inorganic materials with many potential and current applications ranging from environmental remediation and catalysis to drug delivery[1]. Para-aminosalicylate (PAS), a drug used in the treatment of tuberculosis, was intercalated in three different layered double hydroxides (LDH’s): MgAl, ZnAl and CaAl.

LDHs are based on the structure of brucite (Mg(OH)₂), where some of the divalent cation have been substituted by trivalent cations inducing a positive charge on the layers. Anions are intercalated in the interlayers for charge balance. Their general formula is M(II)₁₋ₓM(III)ₓ(OH)₂Ay·nH₂O, where M are metal ions and A an anion[2], 0.16 ≤ x ≤ 0.33. However, intercalation of drugs in LDHs often results in many stacking faults, which render their characterization by PXRD difficult. SSNMR has proven useful to study the cation ordering in the cation layer[3-4], the mobility of the interlayer anions[5], and especially to probe the intercalated anions[6]. The samples were studied in detail using powder X-ray diffraction, elemental analysis, and IR in order to gain insight in to the bulk properties of the sample. Multi nuclear solid state NMR (SSNMR) provided detailed insight into atomic structure of the LDHs. Intercalation of para-aminosalicylic acid in MgAl, ZnAl, and CaAl LDH’s was confirmed by the use of the several techniques including various SSNMR experiments, PXRD and IR. The study shows illustrate the complementary nature of PXRD and SSNMR especially in studies of poorly crystalline materials.

Steel used for engineering design purposes are polycrystalline aggregates consisting of large quantities of individual grains. The steel aggregates are subjected to macroscopic deformation when processed e.g. into plates by rolling. On the microstructural level the deformation manifests itself, among other things, as the grains rotating towards preferred crystallographic orientations. This is also known as the texture of the material. The evolution of the texture is a key area of investigation for metallurgists, as it is a controlling parameter for the mechanical strength of the material and necessary knowledge for the engineering design process.

In this study the lattice rotations of more than 300 individual grains in a 0.7x0.7x0.5 mm$^3$ volume of an austenitic 316 stainless steel sample deformed in tension to 5% elongation have been monitored by 3DXRD microscopy at Cornell High Energy Synchrotron Source (CHESS). The initial grain morphologies and microstructure were also mapped out in high spatial resolution using a near-field detector. This map serves as the input to finite-element based crystal plasticity simulations approximating the measured crystallographic neighbourhood of individual grains closely. The experimental data are compared to the model results, revealing a larger spread of the experimental data. Representative grains are selected for detailed studies to investigate this in more detail.
Synthesis and reduction of iron oxides to α-iron

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Magnets have been around in our daily life since the introduction of commercial magnets in 1730. A quick look around us shows, how much we have integrated the use of magnetism in our everyday life from complex electronic devices such as the computer, loudspeaker etc., to electric motors in cars, wind turbines and to simple magnets used on refrigerators.

The best magnets today are the NIB-magnet, made from neodymium, iron and boron. In general, the best performing magnets are made of rare-earth elements, REE. They are not rare compared to other elements, but they are difficult and expensive to mine and process, which places them in the high-risk zone of being unavailable in the future. Another factor is the geological placement of these REE reservoirs, mainly placed in China, which have led to a severe rare-earth element crisis in the last decade due to a more or less complete monopole of exporting REEs. [1,2]

The aim of the project is to increase the general knowledge of super magnets based on iron nitrides made from pure iron through reducing iron oxides such as magnetite, ferrihydrite or hematite. To do this, the iron oxides are made in steel wrapped Teflon containers to synthesize nanoparticles. These nanoparticles are reduced with hydrogen gas to pure iron nanoparticles, which then in theory can be nitrided with ammonia to produce iron nitrides. The idea of producing iron nitrides is due to iron nitrides being soft magnets, which can be combined with hard magnets to form an exchange coupling, making a super magnet without any rare-earth elements involved. Iron nitrides comes in several forms with the most interesting formulas being the stable γ-Fe₄N reported to reach 160 emu/g and the metastable α″-Fe₁₆N₂, which could reach an astronomical saturation magnetization of 315 emu/g. [3]

The prepared samples are made with different synthesis parameters such as temperature, incubation time, molar ratio of precursors, solvent used to dissolve, adding of base and adding of additional precursors. The composition of the produced particles is measured with PXRD and the particle size is calculated with Rietveld Refinement.

Magnetic short range ordering in frustrated magnets

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Magnetically frustrated materials are gaining a huge increase in interest due to exotic physical phenomena found in spin liquids and glasses. In order to obtain a better understanding of such magnetically disordered materials, magnetic diffuse neutron scattering can be measured and analyzed. For a long time, analysis mainly consisted of inspection of the wave-vector and temperature dependence of scattering, giving only limited information about the disorder. Recently, more advanced methods have been developed, such as modelling the scattering using reverse Monte-Carlo simulations for both powder and single-crystal data. Another recent approach has been to develop a magnetic pair distribution function analysis for powder neutron scattering. Such analysis gives a one-dimensional look at magnetic pairwise interactions, both ordered and disordered.

Here we show a new technique for single-crystal magnetic diffuse scattering analysis. The technique gives a real-space view of the spin-spin correlation in 3D.

We demonstrate the technique on a frustrated magnetic transition metal oxide. Data has been measured at the new instrument CORELLI at the Spallation Neutron Source, Oak Ridge national lab [2]. CORELLI allows for fast and accurate measurements of large volumes of reciprocal space with elastic/inaelastic energy discrimination of unpolarized neutrons [2]. The analysis reveals that in the example compound nearest neighbor metal atoms tend to prefer antiparallel alignment, next-nearest neighbors parallel alignment etc. and that correlations exist up to relatively long distances (> 10 Å).


Direct growth of HKUST-1/graphene composite films on glassy carbon

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Copper-based HKUST-1 is one of the most investigated metal-organic frameworks (MOFs), forming a highly porous lattice with a BET surface area as high as $\sim 1500 \text{ m}^2 \text{ g}^{-1}$. Despite being an insulator itself, this material has been studied for applications in electrochemistry such as CO$_2$ reduction$^{1,2}$ and hydrogen evolution.$^3$ The goal of this research was to develop a general procedure for synthesising stable conductive MOF-based coatings for electrocatalysis. Graphene was mixed with the MOF precursors to form a well-dispersed composite material. The composite coatings were chemically attached to the glassy carbon electrode by means of diazonium electrografting.$^4$ Graphene as additive promoted the growth of films of better quality in terms of coverage and reproducibility compared to bare HKUST-1 coatings. Graphene also restored the conductive properties of the films despite of the blocking MOF layer. The electrochemical stability of the electrode films was also verified.

Left: Optical microscopy images (a, c) and SEM images (b, d) of HKUST-1 and HKUST-1/graphene films grown on glassy carbon plates. Right: Cyclic voltammetry studies of the film electron transfer properties using Ferrocene as redox probe.

Controlling residual stresses in industrial metal components

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Residual stresses are central to the strength, life time and processability of metal components. Moreover, residual stresses in the surface may promote or prevent corrosion, crack formation and environmental stress cracking e.g. from hydrogen embrittlement. In most metal working processes undesired residual stresses are formed. These stresses span over multiple length scales; from the macroscopic stresses to the stress in the bonding of atoms. To predict and measure stresses with certainty is, thus, of high industrial interest. By understanding the formation and development of residual stresses, these can be tailored to enhance the end-use properties of metal worked components.

However, conventional destructive stress measurement techniques all have shortcomings since no recognized method exist for probing stresses inside the materials. In the scientific community, neutron and synchrotron x-ray diffraction have been used to map out stresses. These methods allow tri-axial determination of the stress tensor and can handle complex-shaped components.

Based on cast iron components from the wind industry, we will demonstrate stress measurements using neutron diffraction from ISIS. The barriers for industrial adaptation of stress measurement using neutrons and synchrotrons will be mapped out and discussed. This also involves the development of industrial standardized procedures for residual stress analysis in partnership with synchrotron- and neutron scattering facilities and industrial collaborators. The work is part of the Danish Industrial Neutron Laboratory.
New developments in the McStas neutron Monte Carlo ray-tracing package

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The McStas neutron ray-tracing simulation package is a versatile tool for producing accurate simulations of neutron scattering instruments at reactors, short- and long-pulsed spallation sources such as the European Spallation Source. McStas is extensively used for design and optimization of instruments, virtual experiments, data analysis and user training. McStas was founded as an scientific, open-source collaborative code in 1997. This contribution presents the project at its current state and gives an overview of lessons learned in areas of design process, development strategies, user contributions, quality assurance, documentation, interoperability and synergies with the McXtrace project. Further, main new developments in McStas 2.3 (April 2016) and McStas 2.4 (expected May 2017) are discussed, including many new components, updated source brilliance descriptions, new tools and user interfaces, web interfaces and a new interoperability with MCNP and other high-energy oriented Monte Carlo codes via the MCPL format.
The e-neutrons.org platform - developing a platform for teaching of neutron scattering using blended learning

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Experimental neutron scattering is conventionally restricted to large-scale facilities. It is imperative to educate future users of these facilities in order to get a better scientific output and to stimulate growth of the community. However, students and future users alike can find it hard to locate material to prepare them on the subject of neutron scattering, since very few of their home institutions teach on the topic.

E-neutrons is an online and freely accessible teaching portal to educate students and scientists who are interested in neutron scattering and related techniques. The platform consists of a course management system and software related to showing interactive teaching material.

The introductory neutron scattering course at e-neutrons features a wiki textbook including theory and exercises with hints and solutions as well as many interactive learning quizzes [1]. Some of the learning quizzes are based on online simulation of virtual neutron scattering experiments which is particularly useful in preparing the students for real neutron scattering experiments [2].

This poster will give an overview of our initial results and experiences with using the platform, as well as give information to initiate discussion on what could improve the e-neutrons project.

Complex superstructures in the co-doped high-$T_c$ superconductor $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4+y}$, observed with both neutrons and X-rays

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The high-$T_c$ superconductivity seen in the cuprates constitutes one of the most fascinating problems in the field of condensed matter physics. One of the earliest found high-$T_c$ superconductors, hole-doped $\text{La}_2\text{CuO}_4$, could be a key to understanding the relationship between crystal structures and the complex interplay of magnetism and superconductivity. This material becomes superconducting from hole-doping, which can be done, e.g., by substituting some of the La with Sr or by intercalating O into the structure, making $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ or $\text{La}_2\text{CuO}_{4+y}$, respectively. The intercalated oxygen in the latter are mobile to low temperatures (<200 K), and tends to favor complex modulated structures.

Previous work on the superstructures in the compound mostly focus on a single or a few specific observed superstructures, e.g. a concommittant 1D structure along the $c$ axis – called staging – or a 3D structure assumed to originate directly from the interstitial oxygen [1-3]. However, with this study we have focused on a larger overview of the large number of superstructures, among them also the previously mentioned.

We have mapped reciprocal space of several good quality single crystals of super-oxygenated co-doped $\text{La}_{2-x}\text{Sr}_x\text{CuO}_{4+y}$ with varied Sr doping $x$, all with remarkably coinciding superconducting transition temperature and Néel temperature, $T_c \sim T_N \sim 40$ K [4-5]. We have used several different neutron and X-ray instruments, and measurements have been done at varying temperatures in order to cover several structural phases.

With this poster, we will present some of our fascinating new overviews of the complex superstructures (an example is pictured below), discussing their importance to further our knowledge of the cuprate superconductors.

Investigation of close-range order phenomena during homo- and heterovalent substitution reactions – A summary of X-ray-absorption spectroscopy studies

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A change in chemical composition of an inorganic crystalline solid is typically accompanied by homovalent or heterovalent substitution reactions. In order to describe these reactions and the corresponding structural changes, most investigators rely on traditional long-range order techniques like single-crystal or powder X-ray diffraction. These well-established methods provide precise information about changes in unit cell parameters and can even locate the structural position of elements of interest.

Unfortunately, the results of these long-range order data is often reduced to the simplest interpretations, namely differences in ionic radii of the reaction-involved components and their differences in bond-lengths to their neighboring ligands.

Here, we present a critical summary of two different substitution reactions investigated by extended X-ray absorption fine structure (EXAFS) spectroscopy and important implications of close-range order data for the interpretation of structural changes during homo- and heterovalent substitutions.

In the first study, P⁵⁺ in monoclinic FePO₄·2H₂O (phosphosiderite) was replaced by As⁵⁺. Both elements, P and As, display the same charge and preferred tetrahedral coordination with oxygen. The incorporation of 0.844±0.014 wt.% As⁵⁺ into the phosphosiderite structure lead to a slight increase in unit cell parameters, but the close range order around As changed dramatically to a strengite/scorodite (orthorhombic Fe(P⁵⁺/As⁵⁺)O₄·2H₂O) like arrangement without forming the individual phases. Similarly, in the second study, 1.9 wt.% As⁵⁺ was incorporated into α-Fe₂O₃ (hematite), which lead to a measurable increase in unit cell parameter c of hematite from 13.7690(7) Å in the absence of As⁵⁺ to 13.7943(5) Å for hematite with the highest As content. Unlike phosphosiderite, hematite does not possess cations tetrahedrally coordinated by oxygen and Fe³⁺ is of different charge. The EXAFS investigations at the As K-edge suggest a local coordination similar to Fe₄As₂O₁₁ (angelellite) and the formation of an angelellite-like cluster which intergrowth along the (210) plane of angelellite with the (0001) plane of hematite.

Although long-range order techniques provide vital crystallographic information, these methods cannot fully characterize structural changes during homo- and heterovalent substitution reactions. Due to averaging the atomic positions within the crystal lattice valuable information can get lost. We therefore strongly plead for a combination of long and short-range order techniques to fully understand the mechanisms operating during a substitutions reaction.
Solid state NMR investigations of North Sea Chalk

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A series of chalk samples from the North Sea and Dalbyover chalk mine have been investigated by solid state $^{29}$Si and $^{27}$Al NMR spectroscopy and powder X-ray diffraction, to identify the silica phases and impurities present. Previous studies have shown, that the chalk sections have potential as hydrocarbon reservoirs, and that the geochemical composition may be of great importance for the reservoir properties [1]. The aim of the study is to identify and quantify the SiO$_2$ polymorphs present in the samples.

In the study powder X-ray diffraction and solid state NMR spectroscopy have been used to investigate the local and global structure of the geological samples. From pXRD, the crystalline phases such as $\alpha$-quartz and the crystalline framework in clay minerals are identified in the samples, as illustrated below. However, the samples contain amorphous phases and impurities which cannot be identified by pXRD, but are detected by SS NMR. $^{29}$Si{H} cross-polarization MAS NMR yields additional information of the different SiO$_2$ environments in the samples.

The content of $\alpha$-quartz in the samples can be determined by $^{29}$Si and $^{27}$Al SS NMR and pXRD. Furthermore, $^{29}$Si and $^{27}$Al SS NMR reveals the presence of amorphous SiO$_2$ polymorphs, such as Opal-CT, not seen by PXRD from the Dalbyover chalk mine samples. $^{29}$Si{H} CPMAS NMR reveals the presence of Q$_3$ silica in the sample from the North Sea, and confirms SiO$_2$ defects in the quartz phase.

Dynamical analysis of Ciprofloxacin intercalated in fluorohectorite

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Natural clay minerals are efficient drug carriers providing for high and long lasting drug concentrations owing to their swelling capacity and ion exchange property\(^2,3\). Therefore, considering the large influence of water on the clay mineral properties\(^4,5\) and consequently on the success of the drug intercalation, quasi-elastic neutron scattering measurements were performed using the IN5 time-of-flight spectrometer located at the ILL (Grenoble, France) on Li-Fluorohectorite exposed to 0%, 33%, 55% and 70% relative humidity (RH). Diffraction patterns uncovered from the data confirms the increasing in layer separation of the clay mineral as function of RH. From the analysis of the Generalized Density of States signal (GDOS) we were able to get insight on how the water is affected by confinement at the different RH.

In addition, thermogravimetric analysis (TGA) combined with Fourier-Transformed InfraRed (FTIR) spectroscopy was used to explore the kinetics of decomposition of the various components of ciprofloxacin intercalated into Li-Fluorohectorite at different pHs. By using the Kissinger’s model the activation energies for the surface water of the clay, the water between the clay layers, the two-fold decomposition of the drug in both free and intercalated cases, and the decomposition of the clay itself were determined. The changes of the activation energy in the free and intercalated cases indicate that the drug becomes more stable when intercalated. Furthermore, the amount of water in the clay as well as the number of water molecules per ion site in the clay layers are found to decrease as the pH value of the intercalation process increases.


Superconducting Topological Materials by Doping of Bi$_2$Se$_3$

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Bi$_2$Se$_3$ and Bi$_2$Te$_3$ have been known for many years for their excellent thermoelectric properties [1]. Recently they were also discovered to be topological insulators, which have a bulk band gap and metallic surface states. The crystal structure consists of stacked quintuple layers, Se(1)-Bi-Se(2)-Bi-Se(1), which are separated by weak van der Waals gaps [2]. Topological surface states have also recently been discovered in superconductors [2-4]. Topological superconductors (TS) have surface states that are topologically protected, therefore robust against disorder [5]. This suggests that the unconventional superconductivity can be realized with low sensitivity to impurities.

Extensive work has been devoted to induce superconductivity in topological insulators by doping with a metallic element. Topological superconductors include Cu$_{0.15}$Bi$_2$Se$_3$, Sr$_{0.065}$Bi$_2$Se$_3$ and Nb$_{0.25}$Bi$_2$Se$_3$ with maximum transition temperatures ($T_C$) of 3.8, 2.9 and 3.2 K, respectively [2-4]. The superconductivity is believed to be due to intercalation of Cu, Sr or Nb into the van der Waals gap of the parent compound of Bi$_2$Se$_3$ (Figure 1). The compounds are found to maintain the rhombohedral crystal system ($R\bar{3}m$ space group) as Bi$_2$Se$_3$, but with an elongated c-axis and this is the only structural evidence of intercalation reported in previous studies.

In this study, high quality crystals are being grown for further investigations of the structure-property relations of M$_x$Bi$_2$Se$_3$. Different crystal growth techniques, melt growth, Stockbarger-Bridgman and chemical vapor transport, are applied to obtain high quality crystals. Studies of the temperature dependence of the intercalation is mapped to determine optimal growth conditions while crystallographic studies are used to provide solid structural evidence for the proposed structures.

HEIMDAL at ESS: A thermal neutron powder diffractometer combined with SANS and imaging - A multiple length scale instrument for materials science

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HEIMDAL at ESS will be a multi length scale neutron scattering instrument designed for studying all length scales of advanced functional materials. The instrument combines high resolution or high speed thermal powder diffraction with SANS and imaging. It uniquely features a cold and a thermal guide for fulfill the diverse requirements for diffraction and SANS. With an instrument length of 158 m, HEIMDAL will take advantage of the high neutron flux of the long pulse at ESS, whilst maintaining a high q-resolution due to the long flight path. The powder diffractometer has a q-range coverage of up to 25 1/Å allowing PDF analysis of total scattering data. With the addition of SANS with a 10 m long collimation section and matching distance from sample to detector, HEIMDAL will be able to cover a uniquely broad length scale within a single instrumental set-up.

The first 15 ESS instruments are moving from accepted design concepts and into mature instrument projects. As the the in-kind contribution agreements between ESS and the partner countries are made, the instrument suite at ESS takes form. At the building site the accelerator, target, and guide halls are being built, and rapid progress is made.

At the conference, we will present the latest developments of the instrument project HEIMDAL, where the combination of state-of-the-art neutron scattering techniques will provide the unique possibility to study real materials, in real time, under real conditions.
Deformation and annealing of metals are routine industrial processes for fabricating metal parts. For optimizing the process as well as product it is crucial to be able to control the induced structural changes. Therefore, we investigate the development of the polycrystalline structure of deformed aluminium during annealing to gain insight into the underlying mechanisms. Through annealing series of high resolution x-ray diffraction images we trace thousands of individual micron-sized subgrains inside deformed grains and measure their growth rates and activation energies. At the same time we map recrystallizing grains by means of dark field x-ray microscopy, a recently developed technique that allow for mapping of bulk polycrystalline structures with 100 nm spatial resolution and an angular resolution of millidegrees.
Recent excavations at the European Spallation Source (ESS) construction site in the Swedish city of Lund revealed evidence of prehistoric agriculture in the region. Among the traces were thousands of charred cereal grains, mainly of barley and emmer, the oldest ones of which date back to the formation of the first farming-based societies by the Funnelbeaker culture during the Neolithic period (ca. 4,000 BC).

Analyzing the genetic makeup of a selection of grains can shed light on their origin and their development under the climate of southern Scandinavia. X-ray and neutron tomography were used to study the structure and morphology of the selected grains. The ones most suitable for extraction of DNA can then be singled out based on these results.

By means of X-ray tomography high spatial resolution of the acquired images can be obtained, while neutron tomography facilitates the distinction of organic material. Simultaneous processing of the different data sets, however, requires their perfect alignment (Figure 1). Subsequently, the two methods can be used complimentarily in order to identify organic parts potentially containing genetic material. This alignment procedure will be discussed in this work.
Ammonia containing compounds are of interest because NH₃ can be considered a hydrogen carrier. Originally the system MgCl₂−NH₃ were considered for indirect H₂ storage.¹ By reacting NH₃ with complex hydrides formation of dihydrogen bonds between B−H···Hδ−N occurs, which may lead to release of hydrogen rather than NH₃. By reaction metal alanates may alternate the decomposition mechanism as observed for the metal borohydrides.²

Two new amine alanates have been prepared by reacting Mg(AlH₄)₂ and Ca(AlH₄)₂ with NH₃. Mg(AlH₄)₂ and Ca(AlH₄)₂ were prepared by previous described methods ³, ⁴ The stability of the amine alanates are examined with thermal analysis and the structures are studied by in-situ SR-PXD.

The reaction of NH₃ with the pristine metal alanates results in formation of amine alanates M(AlH₄)₂-xNH₃ (M = Mg and Ca). Thermal analysis of Mg(AlH₄)₂-xNH₃ reveals release of NH₃ at RT. In-situ SR-PXD of Mg(AlH₄)₂ reveals Bragg reflections of a new compound, which is indexed in an orthorhombic unit cell and initial investigation suggest the space group Pcba. The reflections disappears during heating and no reflections from Mg(AlH₄)₂ or MgH₂/Al are observed after decomposition.

Thermal analysis of Ca(AlH₄)₂-xNH₃ shows multiple releases of NH₃ upon heating. Bragg reflections of Ca(AlH₄)₂ disappears after reaction with NH₃. Reflections will occurs when the sample is allowed to release NH₃ at RT.

In this study, it is found that Mg(AlH₄)₂ and Ca(AlH₄)₂ reacts with NH₃ and forms new amine compounds. These amine alanates release NH₃ at RT. Amine Ca(AlH₄)₂ shows multiple NH₃ releases, which may lead to reversible storage of NH₃.

References

Exploring the ligand binding sites of ionotropic glutamate receptor (iGluR) using neutron crystallography

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Ionotropic glutamate receptors (iGluRs) are key players in the central nervous system (CNS) fast excitatory synaptic transmission. iGluRs are crucial for normal brain functioning in learning and memory formation, but their dysfunction can lead to many kinds of severe diseases from depression and epilepsy to neurodegenerative Alzheimer’s disease [1]. Therefore iGluRs are potential drug targets for a huge variety of neurological disorders.

Binding of L-glutamate (Fig) to the iGluR ligand-binding domain (LBD) triggers the receptor activation: the trans-membrane ion pore of iGluRs opens generating postsynaptic action potential. The structure of LBDs is known, it has been investigated in atomic resolution [2], but the exact hydrogen-bonding network around the ligand and the protonation of the residues in the binding pocket remain unknown. In this project, neutron crystallography will be used to investigate the GluA2-LBD structure to clarify the glutamate binding in detail and to get better picture of glutamate and allosteric modulator binding sites for future structure-based drug-design. Protein is expressed and crystallized in fully deuterated conditions to use the advantageous scattering properties of deuterium nuclei in neutron diffraction experiments.


Flexible 3D tomographic alignment and reconstruction of phase-contrast data

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The introduction of coherent diffraction imaging (CDI) in tomography has allowed the acquisition of projection images with high spatial resolution (5–10 nm range) for relatively large fields of view without the need for imaging lenses or optics. This acquired micro and nano-scale information can be of high-interest in different material science applications especially for energy conversion and storage materials where the performance is determined by their internal structure. In order for 3D tomographic reconstructions to exhibit similar spatial resolutions and quantitative measurement accuracy, a proper projection alignment must be performed before tomographic reconstruction. Phase-contrast projection data presents additional properties that must be taken into account prior to tomographic alignment and reconstruction, such as the presence of background linear terms and wrapping areas characteristic of phase signals. We propose an automatic tomographic alignment algorithm insensitive to phase wrapping and linear background terms, including a linear background term removal and a fine iterative optimization step where all angular and linear alignment terms that define a sample-detector relative orientation can be refined. General or complex projection geometries (including tilt angles and uneven projections distribution) can be resolved by means of iterative algebraic tomographic reconstruction algorithms, such as SIRT, that we have extended to phase-contrast wrapped data with the possibility of excluding defective pixel information such as unpaired phase residuals, avoiding unnecessary unwrapping operations that could be responsible for the introduction of undesired artefacts.

Figure 1: On the left: Spatial resolution enhancement (of a tomographic slice) after fine tomographic parameters optimization (including linear and angular components). On the right: Fourier Shell Correlation (FSC) measurements on a simulated Shepp-Logan phantom at the different stages of the proposed algorithm: Before alignment, after coarse translations alignment (CTA), after phase-ramp removal (PRR) and after the fine parameters alignment (FPA).
Phonon contributions to high-temperature superconductivity in LSCO+O

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The compound La2-xSrxCuO4 (LSCO) is superconducting for 0.05 < x < 0.3 and serves as an interesting model-system due to its relatively simple crystalline structure with only one Cu-O layer per unit cell, which is believed to contain the fundamental aspects of superconductivity. Furthermore, LSCO can be doped with interstitial oxygen to form La2-xSrxCuO4+y (LSCO+O). The extra oxygen serves to stabilize the superconducting phase at T_c ~ 40K, as well as a stripe-like antiferromagnetic phase with coinciding TN = 40K, regardless of strontium content [1,2].

While conventional superconductivity is mediated by phonons, it is generally not believed to be the case for high temperature superconductors (HTSC) such as LSCO. However, studies have shown evidence of strong electron-phonon coupling in superconducting cuprates [3] and in light of these findings, we are currently investigating the phonon contributions to HTSC in LSCO+O with inelastic neutron scattering as the primary experimental probe.

Recently, an anomaly (softening) in the longitudinal Cu-O bond-stretching phonon halfway to the zone boundary was found for optimally doped (x=0.15) LSCO. The anomaly disappears for underdoped and overdoped LSCO, suggesting a connection to HTSC [4]. We repeated the experiment for superconducting, but underdoped in terms of strontium, LSCO+O (x=0.06) and found evidence for the same anomaly. These findings strengthen the hypothesis for a phonon contribution to HTSC and thus warrants further investigation.

Atomic structure in molybdenum oxide nanoparticles

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Molybdenum oxides are a fascinating class of materials, in large part due to their applications in important fields such as energy storage, catalysis and solar energy, but just as much due to a rich structural chemistry. A large homologous series of molybdenum oxides phases (Mo_{n}O_{3n-m}) have been characterized in the solid state, while in solution molybdenum oxides form distinct polyoxometalate clusters that vary according to the solvent properties.

One of the most common and studied molybdenum oxides is MoO₂. While bulk MoO₂ is well characterized, MoO₂ nanoparticles suffer from the “nanostructure problem”, and exhibit a different atomic arrangement in nanostructures compared to bulk structures.

To determine the nanostructure, we have synthesized MoO₂ particles with different sizes ranging from c. 100 nm to c. 2 nm via a simple solvothermal synthesis and studied their structure using total scattering techniques in combination with pair distribution function (PDF) analysis, see Figure 1.

Using a relatively simple model, we were able to describe the structure of the large MoO₂ particles, which feature a local structure distortion. Extending this model allows us to describe the structure of the ultra small particles, and gets us closer to establishing a full understanding of the size/structure relations in molybdenum oxide nanoparticles.

Furthermore, we study the Molybdenum oxide cluster chemistry both ex-situ and in-situ with the goals of understanding the formation of the nanostructures, as well as the origin of the local structure distortion.

Figure 6: Pair distribution functions of Molybdenum oxide particles of varying size
An optional focusing SELENE extension to conventional neutron guides: A case study for the ESS instrument BIFROST

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Several of the instruments concepts for the European Spallation Source, currently under construction in Lund, are expected to be up to 165 m long due to its long pulsed structure. Furthermore there is an increased interest in performing experiments in complicated sample environments using high pressures, high or low temperatures and large magnetic fields. The combination of the two requirements imposes a need for specialised neutron optics, that are capable of transporting the neutrons over long distances but at the same time also able to focus the neutron beam to match the experimental conditions.

We here present a new design idea for beam delivery, where a 165 m ballistic guide system with good transport properties is followed by a 4-8 m SELENE guide system similar to Montel optics used for X-ray optics. We have investigated the system by detailed Monte-Carlo simulations using McStas. We show that the background is significantly reduced since the guide system is able to focus the beam almost perfectly on to samples sizes in the range of 0.1-2 mm, while still having a brilliance transfer of 20 - 60% for neutrons of wavelength 4 Å and above.

The project is intended as a case study for the BIFROST instrument, but we argue that this guide system in general can be useful as an optional guide insert when small samples are used in the vicinity of bulky sample environment, e.g. for high-field or high-pressure experiments.
Beyond Hydrogen Storage, Complex Hydrides as Solid Electrolyte for Battery

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The battery safety is a serious concern especially for large-scale applications owing to the utilization of flammable organic liquid electrolytes. Replacing liquid electrolytes by a solid electrolyte will overcome the safety issue.[1] Therefore, identification of solid electrolyte with ionic conductivity comparable to organic liquids is the major scientific challenge.

Recently, complex hydrides were discovered as a new group of Li-ion conductors, e.g. based on BH₄⁻, NH₂⁻, BₙHₙ²⁻. They have light weight, high chemical stability, negligible electronic conduction, and more importantly, complex hydrides (e.g. LiBH₄) form stable and reversible interface with Li metal.[2] Here, we will present the discovery of a superionic phase in the class of lithium amide-borohydrides, Li(BH₄)₁₋ₓ(NH₂)ₓ [3]. The ionic conductivities achieve up to 6.4×10⁻³ cm⁻¹ near room temperature, comparable to lithium ion conductivities of common organic liquid electrolytes (Figure 1). An Li₄Ti₅O₁₂ half-cell based on such an electrolyte displays >60% capacity retention at 3.5 mA/cm² (5C) and stable cycling for 400 cycles.

![Figure 1](image)

Figure 1. a) Arrhenius plots of the ionic conductivity for Li(BH₄)₁₋ₓ(NH₂)ₓ. b) Li-ion conductivity vs. activation energy for Li(BH₄)₁₋ₓ(NH₂)ₓ at 40 °C with values of other lithium solid-state electrolytes reported in literature.