Danscatt 2024 - Program

Aarhus University

Thursday May 30th

11.30 – 12.15	*Registration and lunch* *MatLab, building 1536, room 114*
12.15 – 12.20	**Welcome by Bo Brummerstedt Iversen, AU** *Auditorium E, Mathematics, Building 1533-103*
Session 1	Chair Dorthe Ravnsbæk, AU
12.20 - 12.50	**Ian Robinson, BNL** Origin of the Enhanced Dielectric Response of Ferroelectric Materials seen by Coherent X-ray Scattering
12.50 – 13.15	**Henning Friis Poulsen, DTU** Understanding the physics of metal plasticity using Dark Field X-ray Microscopy
13.15 – 13.45	**Diling Zhu, SLAC** Science at the LCLS X-ray Free Electron Laser
13.45 – 14.10	**Sara Lopez Paz, KU** Mixing anions in van der Waals magnets
14.10 – 14.25	**Kristoffer Støckler, AU** Models of polaron fluctuations in $LuFe_2O_4$
14.25 – 15.00	*Coffee break - **Posters to be displayed in MatLab*** *Building 1536, room 114*
Session 2	Chair Michael Gajhede, KU
15.00 – 15.30	**Alex Evilevitch, Lund University** Temperature-induced DNA density transition in phage lambda capsid revealed with contrast matching SANS
15.30 – 15.55	**Anders Bach, KU** Targeting protein-protein interactions by fragment-based drug discovery to reduce oxidative stress and inflammation
15.55 – 16.20	**Camilla Gottlieb Andersen, AU** Using X-ray crystallography to study monosaccharide partitioning in plants
16.20 – 16.30	**Danscatt PhD Prize announcement**
16.30 – 16.50	**Lecture by PhD prize winner**
16.50 – 17.10	**Lecture by PhD prize winner**
17.10 – 19.00	**Poster session** *MatLab, building 1536, room 114*
19.00	*Dinner Mathematics Canteen*

Friday May 31st

Session 3	Chair Henning Friis Poulsen, DTU
9.00 – 9.30	**Arkadiy Simonov, ETH** Disorder in single crystals: what is it good for?
9.30 – 9.55	**Kim Lefmann, KU** Better neutron instrumentation leads to new discoveries in quantum materials
9.55 – 10.10	**Camilla Buhl Larsen, PSI** Orientation-dependent grain level 3D strain evolution in oligocrystalline CoNiGa
10.10 – 10.25	**Case van Genuchten, GEUS** Using synchrotron-based X-ray techniques to understand the upcycling of arsenic-rich groundwater treatment sludge
10.25 – 10.55	*Coffee break* *MatLab (Building 1536, room 114)*
Session 4	Chair Henrik Birkedal, AU
10.55 – 11.20	**Jeppe Vang Lauritsen, AU** X-ray Photoelectron Spectroscopy in Near-Ambient Pressure Conditions
11.20 – 11.45	**Mads Ry Vogel Jørgensen, AU/MAX IV** *DanMAX – materials science with diffraction and imaging*
11.45 – 12.00	**Estrid Naver, DTU** *Understanding and modelling phase-contrast neutron imaging – is it really just the same as X-ray phase contrast?*
12.00 – 12.15	**Olivia Aalling-Frederiksen, KU** *Structural changes of NiFe-LDH during OER*
12.15 – 12.30	**Report from the chairman - Martin Meedom**
12.30	*Sandwiches and departure / All posters removed* *MatLab (Building 1536, room 114)*

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Origin of the Enhanced Dielectric Response of Ferroelectric Materials seen by Coherent X-ray Scattering

Ian Robinson
London Centre for Nanotechnology, University College, London and
Brookhaven National Laboratory

The dielectric constant of nanoparticle Barium Titanate (BTO) performed three times better than macroscopic materials in Multilayer Ceramic Capacitors, which make use of these leadfree dielectric materials. Various models have been proposed for the enhancement. Here we use Bragg Coherent Diffraction Imaging (BCDI) to understand the "microstrain" defined by the classical Williamson-Hall analysis of neutron or X-ray powder diffraction data. While classical XRD shows the material is cubic, X-ray pair distribution function measurements clearly show the local structure is lower symmetry than cubic. 3D BCDI of selected nanocrystals, shown in Fig 1, reveals the existence of ~50 nm- sized domains, interpreted as tetragonal twins, which cause the average crystalline structure to appear cubic [1]. The ability of these twin boundaries to migrate under the influence of electric fields explains the dielectric anomaly for the nanocrystalline phase.

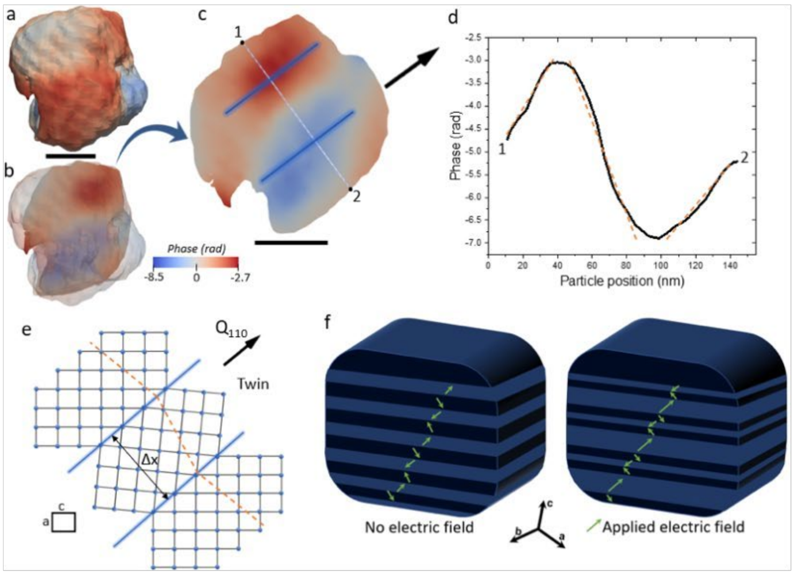


Figure 1. Twin domain model of $BaTiO_3$ (BTO) nanocrystal structure from the Bragg Coherent Diffraction Imaging analysis. (a-c) 3D reconstruction (d) Phase line plot along the white line shown in (c). (e) Schematic of twin domains with the c/a ratio exaggerated ten times (f) Schematic model of the dielectric response as field-induced migration the domain walls.

[1] Ana F. Suzana, Sizhan Liu, Jiecheng Diao, Longlong Wu, Tadesse A. Assefa, Milinda Abeykoon, Ross Harder, Wonsuk Cha, Emil S. Bozin and Ian K. Robinson, Advanced Functional Materials 2208012 (2023)

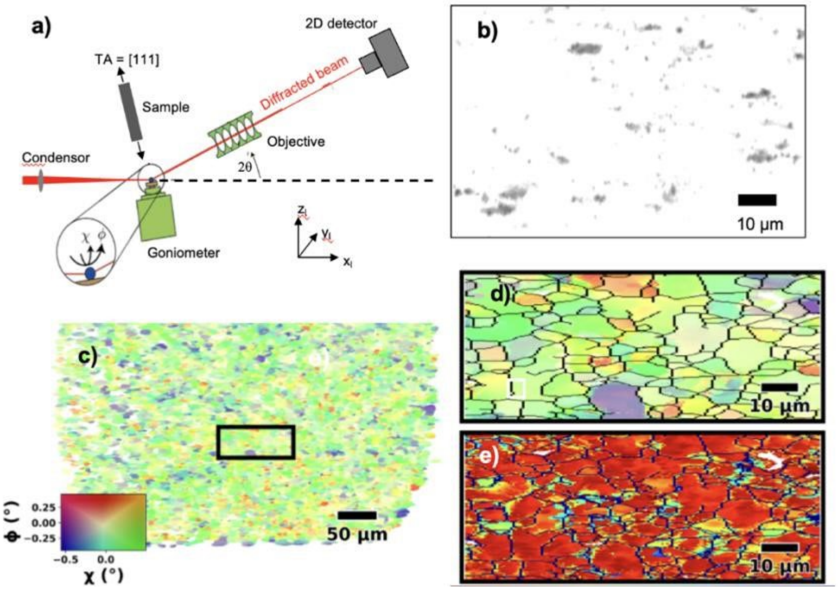
Understanding the physics of metal plasticity using Dark Field X-ray Microscopy

Albert Zelenika, Adam Cretton, Felix Frankus, Sina Borgi, Flemming Grumsen, Can Yildirim, Carsten Detlefs, Grethe Winter, Henning Friis Poulsen

During plastic deformation of metals, trilllions of linear defects (dislocations) are created. These self-organize in cells, which subsequently continuously decrease in size. How and when these processes take place governs the mechanical properties but has remained elusive, because observations of the structural dynamics in the bulk is not feasible with conventional microscopy. As a result, state-of-the-art mechanical modelling has failed rather spectacularly in predicting the patterning, which in turn implies that metallurgy remains a phenomenological science. To overcome this limitation and enable a physics based model of deformation, we have established **Dark Field X-ray Microscopy** (DFXM). Implemented as a flagship beamline ID03 at ESRF, the method provides 3D movies of grains, cells, dislocations and local stress within mm sized specimens – with a spatial resolution of 100 nm.

We here present DFXM movies of the structural evolution during tensile deformation of an aluminium single crystal. The formation and subsequent development of 40,000 cells are visualised. We reveal that cells form in a stochastic and isotropic manner already at 1% strain. We show that the cell size and dislocation density distributions are log-normal and bi-modal Gaussian distributions, respectively, throughout. This insight leads to an interpretation of the formation and evolution steps in terms of universal stochastic multiplicative processes. We complement these results by 3D movies of the interaction of individual dislocations and present how we interface results to 3D modelling.

We present limitation of the method and how we interface the 3D movies to 3D modelling. In outlook we discuss options for improving the instrumental specifications and for performing DFXM studies of irreversible phenomena at XFELs at the 1 µs time scale – to follow the diffusion of dislocations in real time.



a) Sketch of DFXM set up. d) Raw image revealing dislocations and dislocation entanglements c-e) orientation, cell and dislocation density maps at 4.6% deformation. The region-of-interest in d) and e) is marked by a box in c).

Science at the LCLS X-ray Free Electron Laser

Mike Dunne (presented by Diling Zhu)

LCLS, SLAC National Accelerator Laboratory, Standford University

Around the centennial of the discovery of x-rays, development to generate brighter and brighter x-rays has entered a golden age world wide. In particular, the advent of x-ray free electron lasers brought a billion fold leap forward in peak brightness, and ushered in a new era where ultrafast coherent x-ray probes can directly interrogate ever more complex matter at unprecedented timescales. This talk will provide an overview of the Linac Coherent Light Source X-ray FEL at SLAC, including the development of new source capabilities and the overall facility roadmap under consideration. A collection of science application examples spanning material science, chemistry, biology, and high energy density science, will also discussed.

Mixing anions in van der Waals magnets

Sara A. López-Paz
Inorganic Section, University of Copenhagen salp@chem.ku.dk

Two-dimensional (2D) materials displaying intrinsic long-range magnetic order hold the promise to open up new avenues for the electrical manipulation of spins in next-generation quantum technologies [1,2]. In the search for new 2D magnetic materials, mixed-anion chemistry stands as a promising route for optimizing and accessing new magnetic states in van der Waals magnets. As a prime example, CrSBr combines a high magnetic critical temperature ($T_N \approx 140$ K), exotic magneto-transport properties, and complex mixed-anion chemistry[3,4]. We will review the temperature dependent magnetic properties of this material, with special emphasis on the presence of a low temperature hidden order ($T^* = 40$ K $< T_N$) within the long-range magnetically ordered state, and the challenges on the characterization of this elusive order.

References

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3. Ziebel, M. E. *et al.* CrSBr: An Air-Stable, Two-Dimensional Magnetic Semiconductor. *Nano Lett* **17**, 42 (2024).
4. López-Paz, S. A. *et al.* Dynamic magnetic crossover at the origin of the hidden-order in van der Waals antiferromagnet CrSBr. *Nature Communications 2022 13:1* **13**, 1–10 (2022).

Models of polaron fluctuations in $LuFe_2O_4$

Kristoffer Andreas Holm Støckler[1], Nikolaj Roth, Anders Agentoft Feidenhans'l[2], Seiya Takahashi[3], Eiji Nishibori[3], and Bo Brummerstedt Iversen[1]

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Above its charge ordering temperature of 320 K, $LuFe_2O_4$ becomes disordered with local order indicated by the emergence of diffuse helical scattering in its single crystal x-ray scattering patterns. Here we use 3D-ΔPDF informed Monte Carlo modelling to analyse the diffuse scattering in terms of correlated atomic displacements arising from polarons in the charge-disordered system. The local structure of Lu displacements draws conceptual parallels to the classical models of triangular Ising antiferromagnets with the added complexity of interactions between adjacent Lu planes along the crystallographic [001] direction. Extending the models to include displacements of Fe and O results in an excellent description of the experimental 3D-ΔPDF as shown in Fig. 1. By analysing the simulated models, we expose aspects of the hidden order of the Fe(II)/Fe(III) valences in the disordered high temperature state of $LuFe_2O_4$. Particularly, we find a strong correlation between the distortion of trigonal bipyramidal coordination and the bond valence sum estimated charges of Fe indicating a Jahn-Teller polaronic character of the charge carriers in the system [1].

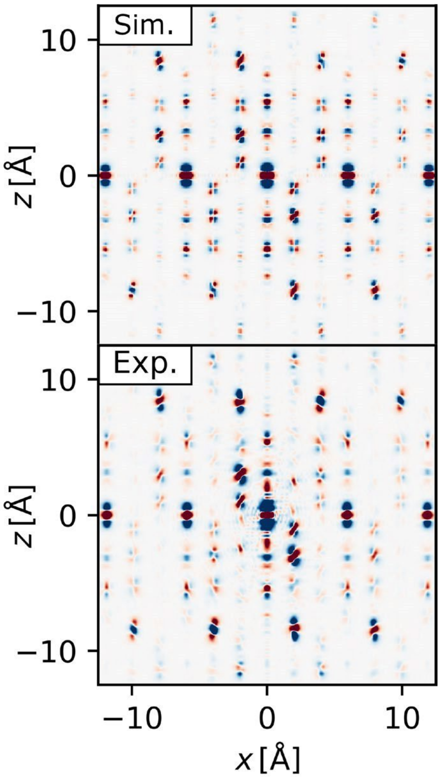

Figure 1. Simulated (top) and experimental (bottom) 3D-ΔPDF of $LuFe_2O_4$ in a representative plane of Patterson space.

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Temperature-induced DNA density transition in phage lambda capsid revealed with contrast matching SANS

José Ramón Villanueva Valencia[1], Efthymios Tsimtsirakis[1], Susan Krueger[2] and Alex Evilevitch[1,*]

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ABSTRACT

Structural details of a genome packaged in a viral capsid are essential for understanding the mechanisms of viral genome packaging and release during the infectious cycle. Furthermore, structural transitions that occur during viral genome uncoating critically influence virus replication dynamics. However, decades of structural virology studies have focused mainly on viral capsids due to their symmetry, rather than on virally encapsidated genomes, where lack of symmetry prevents high-resolution analysis. In this work, we use small-angle neutron scattering (SANS) to reveal the scattering form factor of dsDNA packaged in phage λ capsid by contrast matching the scattering signal from the icosahedral viral capsid with deuterated buffer. Our SANS data shows, for the first time, a temperature-dependent structural transition of intracapsid DNA occurring between two coexisting phases – a hexagonally ordered DNA density in the capsid periphery and a disordered DNA density in the core. As temperature is increased from 20°C to 40°C, we found that core-DNA density increases by ~5 fold, triggering a structural transition in the core-DNA close to the physiological temperature of infection (~37°C). This data explains the structural origin of the previously found temperature-induced, solid-like to fluid-like mechanical transition of packaged λ-genome that facilitates rapid DNA ejection events from phage into a host bacterial cell, causing infection.

Targeting protein-protein interactions by fragment-based drug discovery to reduce oxidative stress and inflammation

Anders Bach

University of Copenhagen

The production and elimination of reactive oxygen species (ROS) are tightly regulated to prevent damaging oxidative stress and maintain redox homeostasis. However, in many diseases, ROS play a central role, for example by inducing inflammation or cellular degeneration. To generate new chemical probes and drug leads we target protein-protein interactions involved in oxidative stress by using fragment-based drug discovery (FBDD). In this talk, I will present the development of our fragment-based drug discovery (FBDD) platform and show recent results related to our main target, the adaptor protein Keap1, which regulates the endogenous antioxidant response via Nrf2.

Using X-ray crystallography to study monosaccharide partitioning in plants

Camilla Gottlieb Andersen

Department of Molecular Biology and Genetics - Protein Science

Aarhus University

Carbohydrates are the most abundant source of energy among all species, which in plants are maintained through photo-driven carbon assimilation. Plant carbohydrate partitioning is sustained by an intricate network of proton motive force coupled transport proteins. Sugar transport proteins (STPs) are responsible for proton-driven uptake of hexose into symplastically isolated cells. We have used X-ray crystallography to structurally characterize two STPs in different conformational states, which has led to defining the underlying transport mechanism used to drive monosaccharide partitioning in plants. Accompanied by biophysical assays we have identified regions that can be used to modulate affinity-induced specificity of STPs, creating a framework for engineering of STP-governed processes in the future.

Disorder in single crystals: what is it good for?

Arkadiy Simonov
Department of Materials, ETH Zürich

Single crystals are known to be the most ordered state of matter. They consist of atomic motifs, the unit cells, which are repeated in three dimensions and tile the entire available space. Most of the properties which we associate with crystals, like the well defined faceted shapes or dielectric anisotropy, or birefringence, are the manifestations of this order. Disorder, is by definition the deviation from this order, and is usually associated with bad crystals.

In this talk I will show that disorder can be incorporated in single crystals without sacrificing their high quality. Moreover, disorder is beneficial for some functional materials. I will show how to precisely characterise disorder using single crystal diffuse scattering, and three dimensional difference pair distribution function, and how it can be used to tune the materials properties like porosity and even manipulate crystal symmetry.

Better neutron instrumentation leads to new discoveries in quantum materials

Kim Lefmann
Niels Bohr Institute, University of Copenhagen

Quantum materials are in general understood as materials where their properties are governed by quantum effects - in particular quantum correlations between electrons. Neutron scattering plays an important role in the investigation of quantum materials by measuring their magnetic structure and dynamics. However, the magnetic moments are often weak and the inelastic signal weak and diffuse, making the measurements and their interpretation difficult. For this reason, development in neutron sources and instrumentation is of great benefit for the understanding of quantum materials. In my presentation, I will present selected examples from our study of quantum magnets, frustrated magnets, and unconventional superconductors and demonstrate the effect of using new neutron sources and instruments. I will also try to predict which effect the BIFROST spectrometer at ESS will have in this field.

Orientation-dependent grain level 3D strain evolution in oligocrystalline CoNiGa

Camilla Buhl Larsen[a], Stavros Samothrakitis[a], Robin Woracek[b], Efthymios Polatidis[a,c], Jan Capek[a], Michael Tovar[c], Søren Schmidt[b], Markus Strobl[a,e]
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Understanding the microstructure of polycrystalline and oligocrystalline materials, in terms of the size, position, and orientation of their individual grains, is integral for enhancing our understanding and potential improvement of their mechanical and functional properties. Three-dimensional neutron and X-ray diffraction tomography methods have both proven efficient at providing grain-scale morphology reconstructions of polycrystalline samples [1-5]. In addition, monochromatic X-ray tomographies have been applied for in-situ strain mapping by tracking how the diffraction spots in a tomography move as stress is applied [6], thereby providing important information on the deformation-behavior of the system.

Here, we present results on grain-resolved strain mapping performed based on Laue three-dimensional neutron diffraction tomography (Laue 3DNDT). Neutrons are more efficient at penetrating bulky samples made up of the elements typically seen in functional alloys, and the use of the white beam both increases the overall experimental efficiency and makes it possible to detect phase transitions and grain re-orientations within a single projection in the tomography. The underlying analysis is based on our novel Laue 3DNDT approach [7-8], in which the initial indexing of the undeformed state is performed using a forward-modelling approach. Subsequent stress states, obtained via in-situ loading of the sample, are then analyzed by fitting grain-resolved strain tensors congruent with the observed diffraction spot movements. We will also discuss the challenges in the analysis related to the utilization of a white beam.

Experimental data using this novel strain mapping method will be presented featuring a CoNiGa sample, which has been prepared using hot-extrusion to promote grain growth and a homogenous oligocrystalline microstructure [9]. The CoNiGa sample has been chosen both for its scientific relevance, being a ferromagnetic shape-memory alloy exhibiting strong anisotropic effects, as well as for its suitability to demonstrate the potential of our method, given the size of the sample and the large grains in the mm-range. We will present the evolution of the strain tensor elements per grain as a function of applied compressive stress and compare them to single-crystal expectations.

References

[1]H. F. Poulsen, *doi.org/10.1007/b97884*
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[6]J. Oddershede et al., *doi.org/10.1107/S0021889810012963*
[7]M. Raventós et al., *doi.org/10.1038/s41598-019-41071-x*
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Using synchrotron-based X-ray techniques to understand the upcycling of arsenic-rich groundwater treatment sludge

C. M. van Genuchten[a], K. Wang[a] [a] Department of Geochemistry, Geological Survey of Denmark and Greenland (GEUS)

Approximately 200 million people are potentially exposed to toxic levels of arsenic (As) in groundwater used for drinking.[1] To decrease the negative health impacts from As exposure, water treatment technologies based on As adsorption to iron (Fe) oxides are widely applied in high- and low-income areas.[2] While these treatment methods are effective, they produce toxic As-rich Fe oxide sludge that must be managed as part of routine operation. Currently, this sludge is treated by the water sector as a waste product to be disposed rather a potential opportunity for resource recovery.

At the same time that As-rich sludge represents a disposal issue, As has now been classified as a Critical Raw Material (CRM) in multiple regions (e.g., EU and USA).[3,4] This classification is based on the growing use of As, particularly As(0) metal, in many products needed for the Green Transition (e.g., batteries, alloys, high-speed electronics). However, the global supply of As(0) metal is largely controlled by a single country, leading to critical global supply risks. In addition, current methods to produce As(0) metal from unsustainable mining and concentrate processing are notoriously harmful to the environment.[5,6]

Taken together, the challenges of As-rich groundwater treatment sludge disposal and the need for local sources of CRMs (i.e., As(0)) to offset global supply risks reveal the clear potential to redefine As-rich treatment sludge as a valuable resource. We have recently developed a novel multi-stage chemical process to upcycle As-rich Fe oxide groundwater treatment sludge, yielding a unique poorly-crystalline form of metallic As(0) as the end-product. The use of synchrotron X-ray characterization data along each step of the upcycling process (Figure 1) has been pivotal to understand the chemical processes underpinning the efficiency of As recovery and valorization. This presentation will report key data (e.g., X-ray absorption spectra, pair distribution functions) obtained from several synchrotrons in Europe and North America that elucidate the solid reaction intermediates and end-products of As-rich waste upcycling. These data have been integral to make knowledge-based improvements in the design of sludge upcycling reactors for high- and low-income regions affected by As contaminated groundwater.

1. Podgorski, J. & Berg, M. Global threat of arsenic in groundwater. *Science (80-.).* **368**, 845–850 (2020).
2. Hering, J. G., Katsoyiannis, I. A., Theoduloz, G. A., Berg, M. & Hug, S. J. Arsenic Removal from Drinking Water: Experiences with Technologies and Constraints in Practice. *J. Environ. Eng.* **143**, (2017).
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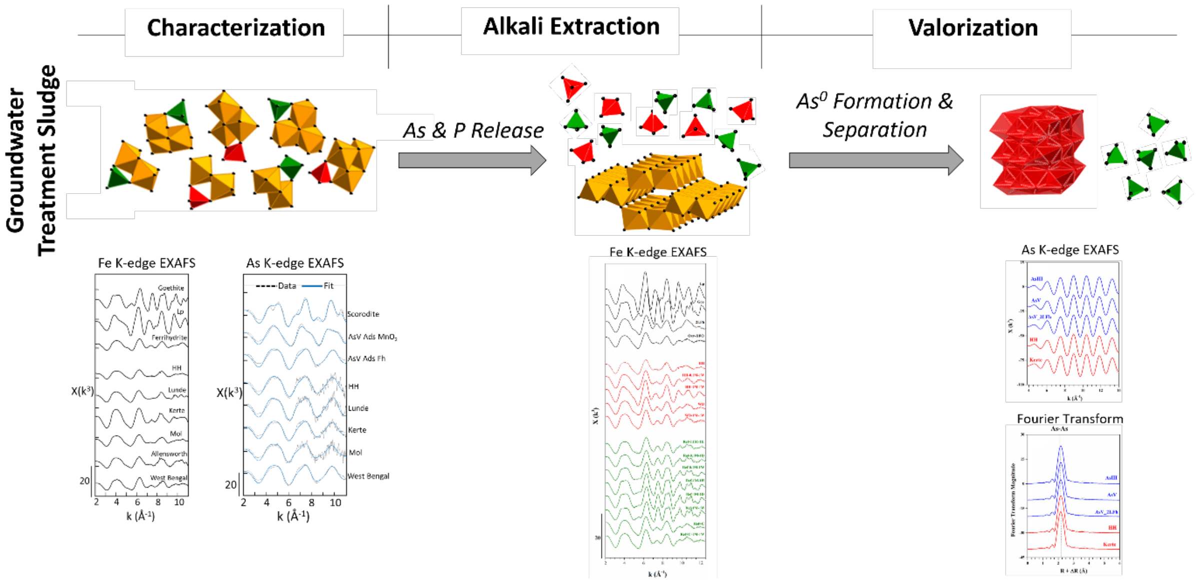

Figure 1: Schematic diagram of the process for upcycling As-rich Fe oxide groundwater treatment sludge, which includes 1) Characterization of the initial sludge, 2) Alkali extraction of surface-bound As and P, and 3) Valorization of the extracted As via selective chemical reduction. Key synchrotronbased X-ray data (Fe and As K-edge XAS) are provided for each step.

X-ray Photoelectron Spectroscopy in Near-Ambient Pressure Conditions

Jeppe Vang Lauritsen
Interdisciplinary Nanoscience Center, Aarhus University

X-ray Photoelectron spectroscopy (XPS) is a widely used technique for surface analysis, providing access to detailed chemical analysis of the topmost layers of solids. XPS is normally restricted to analysis of surface in vacuum. Thanks to development of new analyzers and the use of synchrotron light it has become possible to extend the technique to solid/gas interfaces at relevant pressures, so-called near-ambient pressure (NAP)-XPS. We use this technique in combination with scanning tunneling microscopy (STM) applied in the same pressure conditions, to study planar model systems of heterogenous catalysts. I will show brief examples related to the Cu/ZnO-based catalyst for methanol synthesis and MoS2 catalyst which is used for conversion of bio-oil into renewable hydrocarbon fuels. I will also show how the solid/liquid interface can be probed using NAP-XPS, exemplified by a study of Ni-based electrodes for electrocatalytic water splitting.

DanMAX – materials science with diffraction and imaging

M. R. V. Jørgensen[1,2], I. Kantor[2,3], F. H. Gjørup[1,2], T. E. K. Christensen[3,4]

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The DanMAX materials science beamline is located at the 4th generation storage ring at the MAX IV synchrotron facility in Lund, Sweden. The beamline is designed to be highly versatile and perform both diffraction and tomographic imaging experiments in the energy range 15-35 keV. The very brilliant X-ray source and a flexible optics system allow for three different bandpass modes, ΔE/E ~ 1e-4, 5e-3 & 1e-2, and focusing of the beam from ~10 µm up to ~ 1 mm.

DanMAX currently has two instruments: one for PXRD and one for µCT. Both instruments are designed for *in situ* and *in operando* experiments and are now available to users.

Here, we will present the beamline, the instruments, and some of the initial results from the first years of user operation. We will also briefly present the SINCRYS project, in which we are adding a side station to the beamline. The SINCRYS station will perform singlecrystal X-ray diffraction on micro crystals.

Understanding and modelling phase-contrast neutron imaging – is it really just the same as X-ray phase contrast?

E. B. Naver[1], M. Østergaard[2], D. Battaglia[1], M. Bertelsen[3], P. Willendrup[3,4], P. Trtik[5], O. Yetik[5], M. Strobl[5], S. Schmidt[3], H. Birkedal[2], and L. Theil Kuhn[1]

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Propagation-based phase contrast, for example in the form of edge enhancement contrast, is well established within X-ray imaging but is not widely used in neutron imaging and in many cases avoided. However, this technique can help increase the contrast of low-attenuation materials and decrease noise via phase retrieval [1], [2]. It may confuse quantitative attenuation measurements where the phase signal overlaps the attenuation signal. Therefore, it is important to understand the experimental parameters that cause and affect the edge enhancement in order to optimise experiments to amplify or dampen the effect. In cases where edge enhancement overlaps an attenuation signal, it may be relevant to perform phase filtering to split the phase- and attenuation signals.

We show results from experiments on metal foil samples (Al and Zr) designed for low attenuation and high phase contrast where we have varied the sample rotation, sampledetector distance and neutron wavelength to investigate the effect on the phase signal. The experiments were performed at the ICON and BOA beamlines at the Paul Scherrer Institute [3], [4]. The experiments were compared to simulations based on both the wave theory of the neutron and a particle-based ray-tracing simulation conducted in McStas [5]. Both simulations reproduce attenuation and phase contrast features in the data, but the McStas simulation has a better qualitative agreement with the experimental data.

We also demonstrate phase filtering of experimental data where the attenuation- and phase signals are separated. Two phase-filtering approaches were applied and compared: the transport-of-intensity (TIE) based Paganin filter [1] and McStas simulations of the experiments. The TIE filter worked well at low sample-detector distances, but required a larger than the theoretical value for coherent scattering, which makes this method unsuitable for quantitative phase filtering. The McStas data showed good qualitative agreement between the simulation and experimental data. We present a discussion of the underlying causes and suggest further work on quantitative phase contrast analysis.

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Structural changes of NiFe-LDH during OER

Olivia Aalling-Frederiksen[a], Rebecca K. Pittkowski[a], Gustav K. H. Wiberg[b],
Matthias Arenz[b] and Kirsten M. Ø. Jensen[a]
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Hydrogen production through water electrolysis ($2\ H2O \rightarrow O2 + 2\ H2$) can potentially play a key role in energy storage solutions in the green transition.[1] Here, a major challenge is the production of oxygen, the oxygen evolution reaction (OER), which is a kinetically hindered process with a high overpotential.

Therefore, OER catalyst research is vital. Transition metal (Mn, Fe, Co, Ni) oxides and hydroxides are potential OER catalysts in alkaline media.[2] For instance, transition metal oxyhydroxides with hydrotalcite-like layered double hydroxide (LDH) structures have obtained enormous research attention over the last decades due to their high OER activity.[3, 4] Still, in-depth knowledge about the structural changes of the as-prepared into the catalytically active material is missing.

Here, we investigate NiFe-LDH catalysts under OER conditions using operando XRD and X-ray total scattering with Pair Distribution Function (PDF) analysis, Figure 1. We use a custom-made Gas Diffusion Electrode (GDE) cell which allows material investigation under high current densities and the oxygen evolution phenomena.[5] While the XRD analysis shows how the material undergoes a phase transition with a contraction in the layer-spacing, PDF analysis is used to reveal local structural changes. The PDF analysis shows a contraction of the first atom-atom correlations with material oxidation, hence structural changes within the layers. Our analysis further shows that the phase transition is reversable. However, a surface amorphization is suggested, which clearly affects the extracted crystallite sizes. The change in crystallite size is irreversible.

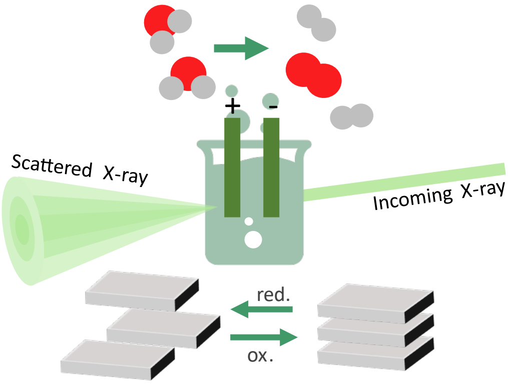

Figure 1 X-ray scattering experiments conducted during the OER showing changes in the layered structure. The reaction is performed in a custom-made GDE cell.

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