Neutrons for Europe
USER MEETING
2022

Oral Presentations in Programme Order

Autonomous neutron experiments

M. Boehm[a], M. Noack[b], T. Weber[a], Y. LeGoc[a], J. Sethian[a], D.E. Perryman[c,a], P. Mutti[a]

[a]Institut Laue-Langevin, Grenoble, France
[b]The Center for Advanced Mathematics for Energy Research Applications CAMERA, Lawrence Berkeley National Laboratory, Berkeley, United States of America,
[c]Grenoble Institute of Technology and Université Grenoble Alpes, Grenoble, France

boehm@ill.fr

Autonomous experiments rely on modern machine learning algorithms for steering experimental data acquisition in a multidimensional parameter space without human intervention. Autonomous data acquisition becomes increasingly important for the execution and analysis of ever more complex experiments in many different scientific and industrial domains [1]. In autonomous learning, algorithms learn from a comparably little amount of input data which future steps to take, in order to explore this multidimensional space in a most efficient way.

In the context of neutron scattering this concept of autonomous experimentation offers alternative ways of data acquisition, especially on single detector instruments with a point-by-point data acquisition. Correlating available information of data points via an algorithm like Gaussian process regression (GPR) [2] can support the acquisition strategy of the experimentalists, by e.g. estimating the posterior mean and covariance to calculate the optimal next measurement point.

We present first results obtained on ThALES, the cold neutron three-axis spectrometer at the Institut Laue-Langevin, using the GPR based algorithm gpCAM [3]. The algorithm explored in an agnostic way, i.e. without any prior information on the physical model, various accessible regions in the sample's reciprocal space and reconstructed sections of S(Q,w) with a considerable reduced number of total measuring points compared to a conventional 'grid' scanning technique. As a next logical step we work on physically informed methods to further increase the efficiency of the measurements.

We try to show a perspective of the future experimental possibilities and how the scientific measurements could evolve in conjunction with modern algorithms.

References

[1] e.g. Noack, M.M., Zwart, P.H., Ushizima, D.M. *et al.* Gaussian processes for autonomous data acquisition at large-scale synchrotron and neutron facilities. *Nat Rev Phys* (2021). https://doi.org/10.1038/s42254-021-00345-y.

[2] e.g. C. E. Rasmussen and C. K. I. Williams, *Gaussian Processes for Machine Learning*, the MIT Press, 2006, ISBN 026218253X. © 2006 Massachusetts Institute of Technology.

[3] https://gpcam.lbl.gov.

Neutrons and Current Health Challenges

Stora Salen

11:00-12:30

Structural investigation of lipid nanoparticles is key for successful mRNA delivery

Marianna Yanez Arteta[a]

Advanced Drug Delivery, Pharmaceutical Sciences, R&D, AstraZeneca, Gothenburg, Sweden

mariann.yanezarteta@astrazeneca.com

The use of messenger RNA (mRNA) as a therapeutic treatment to produce a deficient protein in situ became a reality in 2020: 2 mRNA-based vaccines against SARS-CoV-2, produced by Pfizer/BioNTech and Moderna, received emergency authorization by multiple regulatory agencies around the world. The technology that made this possible was a lipid nanoparticle (LNP) used as a delivery vehicle. These LNPs are mainly composed by a cationic ionizable lipid, which is responsible of encapsulating and facilitating release into the cell cytosol, and helper lipids to provide stability to the particle such as cholesterol, a phospholipid and a poly(ethylene glycol) lipid. Despite the great advances in LNP development, there are still challenges to overcome relating to their efficacy, which is typically in the single digit percentage, as well as ensuring their safety.

Our work shows how elucidation of the structure of mRNA-containing LNPs is used for their rational design that leads to an improvement in their efficacy. Additionally, we have investigated the fate of mRNA-containing LNPs in the presence of proteins Small angle X-ray and neutron scattering (SAXS and SANS, respectively) has allowed us to probe the internal structure of the LNPs as well as the lipid distribution across them. Furthermore, we used SANS to investigate what happens to the structure of mRNA-containing LNPs upon protein binding. We have found that both cholesterol and the phospholipid DSPC are enriched at the LNP surface, giving them a core-shell profile. These results allowed us to variate the LNP size and surface composition in order to increase intracellular protein production of up to 50-folds for certain LNPs in 2 different types of clinically relevant cells, human adipocytes and hepatocytes. This improvement is most likely related to the ability of LNPs to fuse with early endosome membranes[1].

Another important consideration is the formation of a protein corona at the LNP surface upon administration. Apolipoprotein E (ApoE) is responsible for fat transport in the body and has been identified as one of the main proteins in the LNP corona. We found that binding of ApoE to LNPs induces a redistribution of the lipid across the particle, which can impact endosomal escape.[2] Probing the structure of mRNA-containing LNPs with techniques as SAXS and SANS lead us to gain fundamental understanding on the LNP structure. This information provides insight into the mechanisms leading to LNP effectivity and will improve the possibility of success of mRNA therapies.

References

[1] Yanez Arteta, M.; Kjellman, T.; Bartesaghi, S.; Wallin, S.; Wu, X.; Kvist, A. J.; Dabkowska, A.; Székely, N.; Radulescu, A.; Bergenholtz, J.; Lindfors, L. *PNAS* **2018**, E3351-E3360.
[2] Sebastiani, F.; Yanez Arteta, M.; Lerche, M.; Porcar, L.; Bragg, R.A..; Lang, C.; Elmore, C. S.; Krishnamurthy, V. R.; Russell, R.A; Darwish, T.; Pichler, H.; Waldie, S.; Moulin, M.; Haertlein, M.; Forsyth, V.T.; Lindfors, L. and Cárdenas, M. *ACS Nano* **2021**, 15, 6709–6722

Neutrons for structural glycobiology: lectin-carbohydrate recognition in host-pathogen interactions

Lukas Gajdos[a], Matthew P. Blakeley[a], V. Trevor Forsyth[b], Michael Haertlein[a], Juliette M. Devos[a], Anne Imberty[c].

[a]Institut Laue-Langevin, Grenoble, France, [b]Faculty of Medicine, Lund university and LINXS Institute for Advanced Neutron and X-ray Science, Lund, Sweden, [c]Université Grenoble Alpes, CERMAV, CNRS, Grenoble, France

gajdosl@ill.fr

Lectins are carbohydrate-binding proteins that play important roles in cell recognition and host-pathogen interactions. Many pathogenic bacteria produce lectins that are specific for glycans present on the host surface and that participate in adhesion in the early stages of infection. Lectin-carbohydrate interactions are mostly formed by hydrogen bonds between the sugar hydroxyl groups and the amino acid residues of the lectin binding sites. Other types of interactions can also be involved including CH-π stacking, hydrophobic interactions, water-bridging or metal coordination.

Using neutron macromolecular crystallography, we have unravelled the details of protein-carbohydrate interactions in two fucose-specific lectins, with the unique feature of producing perdeuterated monosaccharide fucose using a glyco-engineered strain of *E. coli* bacteria for the preparation of co-crystals [1]. PLL lectin from bacteria *Photorhabdus luminescens* was chosen as a model system for the detailed description of the H-bonding network involved in sugar recognition, including direct and water-bridged hydrogen bonds and CD-π stacking interactions between the apolar face of fucose and aromatic amino acids [2]. LecB lectin from *Pseudomonas aeruginosa,* a human opportunistic pathogen that causes lethal infections in cystic fibrosis patients, is currently viewed as a potential drug target for glycomimetic compounds with antiadhesive properties. LecB displays an unusually high affinity towards fucose with two calcium ions involved in the binding. The neutron study enabled a complete description of the hydrogen-bonding network and the protonation state of charged amino acids involved in the sugar-binding, including the observation of a low-barrier hydrogen bond between fucose and the protein [3]. The new structural data may help in the design of new potent glycomimetic compounds for fighting antibiotic-resistant bacteria.

References

[1] Gajdos, L., Forsyth, V.T., Blakeley, M.P., Haertlein, M., Imberty, A., Samain, E., and Devos, J.M. (2021). *Glycobiology*. **31**, 151–158.

[2] Gajdos, L., Blakeley, M.P., Kumar, A., Wimmerová, M., Haertlein, M., Forsyth, V.T., Imberty, A., and Devos, J.M. (2021). *Structure*. **29**, 1003–1013.

[3] Gajdos, L., Blakeley, M.P., Haertlein, M., Forsyth, V.T., Devos, J.M., and Imberty, A. (2022). *Nature Communications*. **13**, 194.

Neutron crystallography to uncover SARS-CoV-2 main protease function and design potent inhibitors

Andrey Kovalevsky[a]

[a]Oak Ridge National Laboratory, Oak Ridge, TN, U.S.A.

kovalevskyay@ornl.gov

COVID-19, caused by SARS-CoV-2, remains a global health threat after two years of the pandemic even with available vaccines and therapeutic options. The viral main protease (Mpro) is indispensable for the virus replication and thus is an important target for the design and development of small-molecule antivirals. Computer-assisted and structure- based drug design strategies rely on atomic scale understanding of the target biomacromolecule traditionally derived from crystallographic data collected at cryogenic temperatures. Conventional protein X-ray crystallography is limited by possible cryo-artifacts and its inability to locate the functional hydrogen atoms crucial for understanding chemistry occurring in enzyme active sites. Neutrons are ideal probes to observe protonation states of ionizable amino acids at near-physiological temperature, directly determining their electric charges and hence accurately mapping the active site electrostatics – crucial information for drug design. Our room-temperature X-ray crystal structures of Mpro brought rapid insights into the reactivity of the catalytic cysteine, malleability of the active site, and binding modes of various protease inhibitors. The neutron crystal structures of ligand-free and inhibitor-bound Mpro were determined allowing the direct observation of protonation states of all residues in a coronavirus protein for the first time. At rest, the catalytic Cys-His dyad exists in the reactive zwitterionic state, with both Cys145 and His41 charged, instead of the anticipated neutral state. Covalent inhibitor binding results in modulation of the protonation states. This information was used to design nanomolar hybrid reversible covalent inhibitors with robust antiviral properties. High-throughput virtual screening, utilizing available supercomputing capabilities, in conjunction with in vitro assays identified a lead noncovalent compound with sub-micromolar affinity. The neutron structure of Mpro in complex with the noncovalent inhibitor was used in a structure-activity relationship (SAR) study guided by virtual reality structure analysis to novel Mpro inhibitors with improved affinity to the enzyme. Our research is providing real-time data for atomistic design and discovery of Mpro inhibitors to combat the COVID-19 pandemic and prepare for future threats from pathogenic coronaviruses.

Understanding the microscopic properties and drug diffusion kinetics in long-acting peptide hydrogel drug delivery implants for HIV/AIDs.

Garry Laverty[a], Emily R Cross[a], Sophie M Coulter [a], Sreekanth Pentlavalli [a], Yuming An [a], Ralf Schweins [b].

[a] Biofunctional Nanomaterials Group, School of Pharmacy, Queens University Belfast, MBC, 97 Lisburn Road, Belfast, N. Ireland, BT9 7BL
[b] Institut Laue – Langevin, 71 Avenue des Martyrs, CS 20156, 38042 Grenoble Cedex 9, France

garry.laverty@qub.ac.uk

Background: To fulfil the need for a convenient and effective long-acting formulation to deliver drugs to HIV/AIDS patients over a sustained period, we have developed an injectable *in situ* forming peptide hydrogel implant for the delivery of HIV/AIDS drugs for ≥28 days [1]. Our formulation is an enzyme-responsive self-assembling low molecular weight D or L-α peptide hydrogelator, namely phosphorylated (naphthalene-2-ly)-acetyl-diphenylalanine-lysine-tyrosine-OH (NapFFKY[p]-OH), to which zidovudine is conjugated covalently via an ester linkage. This forms a powder that can be readily dissolved in aqueous buffer to create an injectable solution.

Aims/Objectives: To understand the macroscopic properties of gels and the relationship between their underyling structure and the diffusion kinetics of the HIV/AIDS drug zidovudine using primarily small angle neutron scattering (SANS), rheology and drug release assays.

Methods: SANS [D11 ILL,], oscillatory rheology (loss/storage moduli, critical strain, viscosity, time sweeps), scanning electron microscopy, circular dichroism, *in vitro* (phosphate buffered saline) and *in vivo* (Sprague Dawley rats, subcutaneous injection) drug release for ≥28 days.

Findings: Rheology showed peptides demonstrated enzyme-instructed self-assembly, forming hydrogels (~1KPa – 10KPa) within minutes. Drug attached peptide gels were less stiff than gels formed from parent peptide with no drug. SANS showed gels form fibres of narrow radius (~2nm) and large length, a common property of entangled gel fibers [2]. Data closely fitted the flexible cylinder elliptical model and the composition of the gel fibres were similar at low Q. Differences in gel stiffness when zidovudine is attached to the peptide was attributed to entanglement of fibres, rather than the composition of fibres themselves or their secondary structures (primarily β-sheets). The D-peptide variant Napffk(zidovudine)Y[p]G-OH demonstrated zero-order drug release kinetics over 28 days. Clinically relevant plasma levels of zidovudine were detected in rat models for up to 35 days.

Conclusions: A large component of gel strength can be controlled by external conditions and this may allow a change in formulation parameters to optimise material specifications, most notably gel strength and drug release kinetics for long-acting drug delivery.

References

[1] A. Owen, S. Rannard, Advanced drug delivery reviews, 103 (2016) 144-156.
[2] E.R. Draper, B. Dietrich, K. McAulay et al, Matter, 2 (2020) 764-778.

Fundamental and Particle Physics

Stora Salen

13:30-15:00

Nuclear physics research at ILL – an overview

M. Scheck[a], *C. Michelangoli*[b], *U. Koester*[b]

[a]*CEPS – University of the West of Scotland, Paisley, UK*

[b]*Institut Laue-Langevin, Grenoble, France*

marcus.scheck@uws.ac.uk

The talk will introduce the existing nuclear physics research infrastructures at ILL. These are the Fission Product Prompt g-ray Spectrometer (FIPPS) [1] and the LOHENGRIN mass separator with its focal plane equipment [2], and to some degree the multi-purpose intense polarizable cold-neutron beam instrument PF1b. These installations exploit the intense neutron flux from ILL's reactor for neutron-induced reactions such as (n,g) neutron capture and (n,f) neutron-induced fission. For each instrument, the technical outline and the reaction mechanisms are introduced. Furthermore, currently accessible observables are demonstrated by presenting for each instrument a recently published physics highlight. More specifically these are:

A 96-Ytrium to 96-Zirconium b-decay experiment performed at LOHENGRIN investigated one of three top contributors to the reactor antineutrino anomaly. While strong efforts are made to quantify the reactor antineutrino anomaly using highly-efficient setups with comparably low resolution, e.g., see References [3,4], the above-mentioned study represents a first approach to investigate the nature of high-lying final states using high-resolution spectroscopy [5].

A ^{63}Ni(n,g) experiment utilizing FIPPS as part of a campaign at multiple facilities investigated the nature and shapes of excited 0^+ levels in ^{64}Ni. This experiment, which employed the radioactive beam of free neutrons on a 2 GBq radioactive 63-nickel target, was key to identify further levels built upon these 0^+ states [6] and allowed to measure lifetimes and, therefore, transition rates. The obtained detailed experimental picture serves as test case for large-scale shell model calculation, which include the novel mechanism of type-II shell evolution [7]. The latter is a stunning development that considers dynamical shell structures for protons and neutrons, which strongly depend on occupation numbers in excited states of certain orbitals near the Fermi surface.

Additionally, examples of future technical developments, which will extend the already impressive nuclear physics research opportunities at ILL will be presented.

References

[1] C. Michelagnoli et al., EPJA **193**, 04009 (2018)
[2] G. Fioni, et al., Nucl. Instrum. Meth. A **332**, 175-180 (1993)
[3] M. Estienne et al., Phys. Rev. Lett. **123**, 022502 (2019)
[4] D. Adey. et al. Phys., Rev. Lett. **123**, 111801 (2019)
[5] K. Mashtakov et al., Phys. Lett. **B820**, 136569 (2021)
[6] N. Marginean et al., Phys. Rev. Lett. **125**, 102502 (2020)
[7] T. Togashi et al., Phys. Rev. Lett. **117**, 172502 (2016)

Weak and which-way measurements in neutron interferometry (withdrawn)

Stephan Sponar

Atominstitut, TU Wien, 1020 Vienna, Austria

Stephan.sponar@tuwien.ac.at

Neutron interferometry [1], where an interference effects of matter waves passing through a perfect silicon-crystal interferometer is observed, has established a powerful tool for investigation of fundamental quantum mechanical concepts with massive particles. This technique enabled several text-book experiments, such as demonstrations of 4 π spinor symmetry of spin-1/2 particles, spin superposition, gravitationally induced phase and topological phase effect, as well as studies of characteristics of intra-partite entanglement, i.e., entanglements between different degrees of freedom [2]. In this talk I will give an overview of our recent work on weak measurements [3], a new type of quantum variable introduced by Yakir Aharonov in 1988. Our neutron optical approach is realized by utilizing neutron interferometry, where the spin of the neutron is coupled weakly to its spatial degree of freedom [4]. This scheme is applied to study a new counter-intuitive phenomenon, the so-called quantum Cheshire Cat, where a quantum system, subject to a certain pre- and post-selection, behaves as if a particle and its property are spatially separated [5]. In quantum mechanics, observables are usually represented by Hermitian operators, but the product of two non-commuting observables as occurring in the commutator relation is, in general, non-Hermitian. However, the framework of weak measurements allows for a direct test and thus an experimental verification of the canonical commutation relation between two qubit observables [6]. In a recent experiment, we experimentally demonstrate that an individual neutron moving through a two-path interferometer is actually physically distributed between the two paths. The obtained path presence equals the weak value of the path projector and is not a statistical average but applies to every individual neutron, verified by the recently introduced method of feedback compensation [7].

References

[1] H. Rauch, W. Treimer, and U. Bonse, *Phys. Lett. A* **47**, 369 – 371 (1974).

[2] S. Sponar, et al., *Nat. Rev. Phys.* **3**, 309 – 327 (2021).

[3] Y. Aharonov, D. Z. Albert, and L. Vaidman, *Phys. Rev. Lett.* **60**, 1351 – 1354 (1988).

[4] S. Sponar, et al., *Phys. Rev. A* **92**, 062121 (2015).

[5] T. Denkmayr, H. Geppert, S. Sponar, H. Lemmel, A. Matzkin, J. Tollaksen, and Y. Hasegawa, *Nat. Commun.* **5**, 4492 (2014).

[6] R. Wagner, W. Kersten, A. Danner, H. Lemmel, A. K. Pan, and S. Sponar *Phys. Rev. Research* **3**, 023243 (2021).

[7] H. Lemmel, N. Geerits, A. Danner, H. F. Hofmann, and S. Sponar *Phys. Rev. Research* **4**, 023075 (2022)

BeamEDM – A beam experiment to search for the neutron electric dipole moment

Anastasio Fratangelo on behalf of the collaboration

Laboratory for High Energy Physics and Albert Einstein Center for Fundamental Physics, University of Bern, Bern, Switzerland

anastasio.fratangelo@lhep.unibe.ch

The neutron Electric Dipole Moment (EDM) has attracted interest as a promising channel for finding new physics for a long time. The existence of a neutron EDM would violate CP symmetry given CPT conservation. This new source of CP violation could explain the baryon asymmetry of the universe. The BeamEDM experiment aims to measure the neutron EDM using a novel technique which overcomes the previous systematic limitation of neutron beam experiments, the relativistic vxE effect. The experiment exploits the time-of-flight technique with a pulsed cold neutron beam which allows to distinguish between time dependent and time independent effects such as the EDM. A proof-of-principle apparatus has been developed to perform preliminary measurements for the future full-scale experiment intended for the European Spallation Source in Sweden. In this presentation the details of the experimental setup together with the latest results from a data taking campaign in August/September 2020 at the PF1b beamline at the Institut Laue-Langevin in France will be presented.

BRAND – search for exotic couplings in weak interactions using the transverse electron polarization in the decay of free neutrons

Kazimierz Bodek[a] *(on behalf of the BRAND Collaboration)*

[a] *M. Smoluchowski Institute of Physics, Jagiellonian University, Cracow, Poland*

kazimierz.bodek@uj.edu.pl

Neutron and nuclear beta decay correlation coefficients are sensitive to the exotic scalar and tensor interactions that are not included in the Standard Model (SM). The experiment BRAND will measure simultaneously seven neutron correlation coefficients: *H*, *L*, N, *R*, *S*, *U* and *V* that depend on the transverse electron polarization – a quantity that vanishes in the SM. Five of these correlations: *H*, *L*, *S*, *U* and *V* were never attempted experimentally before. The expected impact of this experiment is comparable to that of frequently measured "traditional" correlation coefficients (*a*, *b*, *A*, *B*, *D*) but offers completely different systematics and additional sensitivity to imaginary parts of the scalar and tensor couplings. In order to demonstrate the feasibility of the challenging techniques such as the event-by-event decay kinematics reconstruction together with the electron polarimetry a test program is ongoing on the PF1B beam line at the Laue-Langevin Institute, Grenoble, France (ILL), with simplified experimental setups. The strategy of the project assumes a gradual increase of sensitivity by extending the fiducial volume and angular coverage of detectors. The next step on this way will be to construct a full one meter long detection segment and install it for characterization and initial data taking at ILL. It is planned that the ultimate accuracy will be achieved on the ANNI beam line of the European Spallation Source, Lund, Sweden, with multiple detecting segments. Depending on the experience gained in the proceeding phase, the apparatus may be upgraded with the Mott target made of depleted uranium and, possibly, will be run at reduced pressure in electron tracking detectors.

Diffraction and Applied Materials

ROOM

13:30-15:00

Peter Hedström

Title

Author

Abstract

References

In situ neutron diffraction investigation of grain boundary effect on degradation of superelasticity in near equiatomic NiTi alloy

Zifan Wang[a], Alexander M. Korsunsky[a]

[a]MBLEM Department of Engineering Science, University of Oxford, Oxford OX1 3PJ, United Kingdom

zifan.wang@exeter.ox.ac.uk / zifan.w@outlook.com

To explore the possibility of customising the functional behaviour of NiTi shape memory alloy via controlling the grain structure, binary $Ni_{55}Ti_{45}$ (wt.%) alloys were manufactured in *as cast*, *hot swaged, and hot rolled* conditions [1], presenting contrasting types of grain boundary (GB) and macroscopic performance. *In situ* time-of-flight neutron diffraction technique was employed to study the GB effect on the microstructural evolution during Superelasticity, and a range of properties were evaluated. It was found that (i) hot swaging process leads to change in grain morphology and increase in microstrain; (ii) thermal expansion coefficients of martensite and austenite variants were weakly affected by the texture and phase transformation constraint; (iii) significant GB effect on the elastic properties at both macro- and micro-scale was quantified by Crystal Plasticity Finite Element (CPFE) modelling approach; (iv) texture evolution during Superelasticity is weakly related to the initial microstructure; (v) martensite reoriented so that the <010> axis became aligned parallel to the loading direction, and retained this orientation upon unloading, revealing the underlying correlation between texture evolution and detwinning. Based on the experimental results, an 1D model was proposed to quantify the lattice strain evolution during Superelasticity. Validity of the model equation was confirmed by comparison with experimental observations.

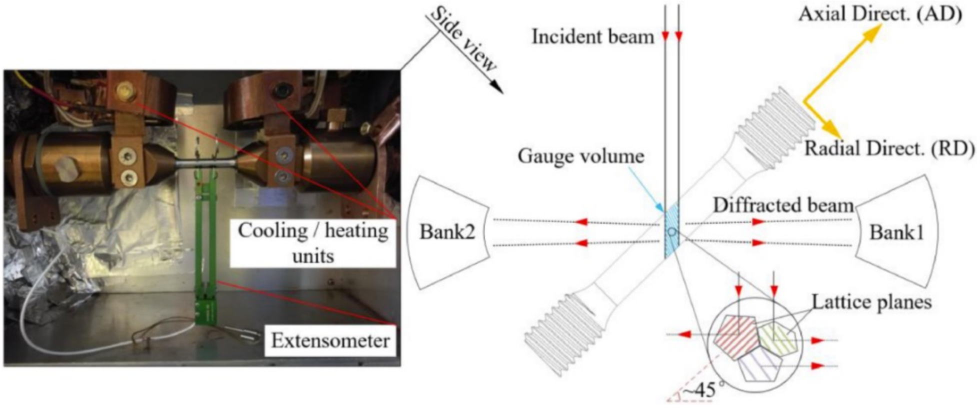


Figure 1. Setup for in situ neutron diffraction experiment on Superelasticity

References

[1] Z. Wang, A.M. Korsunsky, Effect of Temperature on Shape Memory Materials, Encyclopedia of Smart Materials 4, Pages 239-253. https://doi.org/10.1016/B978-0-12-803581-8.11793-X.

Standardization of industrial residual stress measurement for metallic components: diffraction and destructive techniques

Wen Cui [ac], Ranggi S. Ramadhan [b], Sandra Cabeza [b], Philip J. Withers [a], Matthew J. Roy [c]

[a]Henry Royce Institute, Department of Materials, University of Manchester, Oxford Road, M13 9PL Manchester, UK
[b]Institut Max von Laue – Paul Langevin (ILL), 71 avenue des Martyrs, CS 20156 38042 Grenoble, France
[c]Department of Mechanical, Civil and Aerospace Engineering, University of Manchester, Oxford Road, M13 9PL, Manchester, UK.

wen.cui@manchester.ac.uk

This study concerns the European Activity for Standardization of Industrial Residual Stress Characterization (EASI-STRESS [1]) Round Robin activities, aimed at harmonizing measurement techniques over the widest sources of residual stress in modern metal manufacturing. In this work, you would have the chance to get a general overview of EASI-STRESS benchmarking activities and see our preliminary results obtained from neutron and synchrotron diffraction.

Macroscopic residual stresses in metallic engineering components arise due to chemical, thermal and plastically induced misfits and often cause safety concerns and/or reduced lifetime during manufacture and in service. A residual stress field in a manufactured metallic part can be complicated by factors including stress triaxiality driven by geometric features, stress gradients caused by inhomogeneous microstructure, along with localised stress concentration due to rapid change in temperature or chemical compositions [2]. A wide range of techniques, often being classified into destructive, non-destructive, and semi-destructive, are available for estimating residual stress within metals. Hole drilling and Laboratory X-ray are likely the most widespread methods due to their relatively high accessibility and low cost, and thus are preferred by many industries. High energy diffraction techniques (neutron and synchrotron), although bringing new opportunities and capabilities for stress evaluation, are not yet widely adapted by industries. We aim to help industries to gain better understanding of neutron and synchrotron diffraction for stress measurement in comparison to common laboratory techniques, and to strength industrial access to high-energy diffraction methods.

References

[1] easi-stress.eu

[2] PJ Withers, H Bhadeshia, Residual Stress Part 2 – Nature and Origins, 2001. Mater. Sci. Tech., 17 (4), 366–375

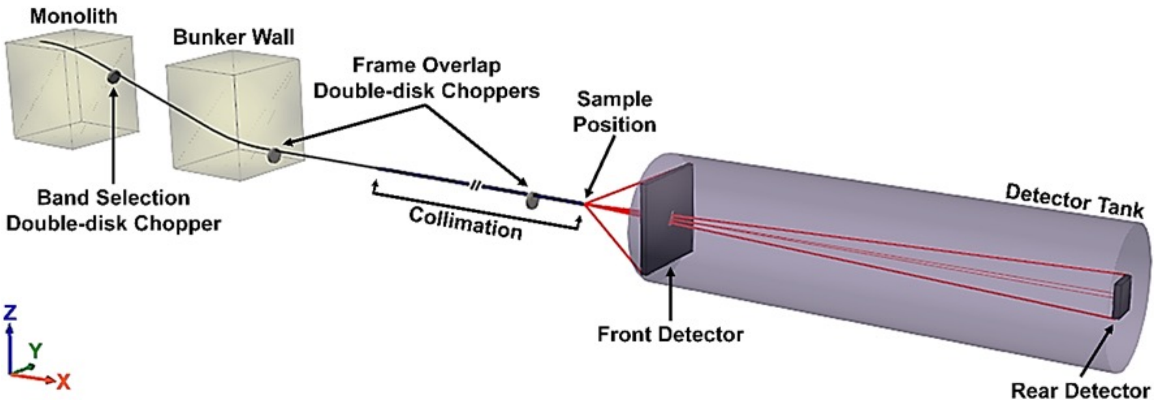
Considering instrumentation for a high intensity moderator at the European Spallation Source

Stavros Samothrakitis[a], M. Bertelsen[b], P. Wilendrup[c], E. Knudsen[c], M. Strobl[a,d]

[a]Paul Scherrer Institute, Villigen, Switzerland
[b]European Spallation Source ERIC, Lund, Sweden
[c]Technical University of Denmark, Lyngby, Denmark
[d]University of Copenhagen, Copenhagen, Denmark

stavros.samothrakitis@psi.ch

The European Spallation Source, currently under construction in Lund, Sweden, will originally make available a suit of 15 state-of-the-art neutron scattering instruments, which will be served by a high-brightness moderator placed above the spallation target. The current infrastructure of ESS, however, allows for a second, alternative moderator to be constructed and positioned below the spallation target. The second moderator is currently considered to have a larger viewable area, offering higher total cold neutron flux and higher intensities at longer wavelengths, potentially spanning a range from Cold (4 – 10 Å) to Ultra Cold (> 100 Å) neutrons. It is assumed that several areas of condensed matter research profit from such second moderator concept, including small-angle neutron scattering, spin-echo spectroscopy, and neutron imaging. Here, we present the conceptual designs of potential instruments, mainly giving focus to small-angle neutron scattering.



References

[1] Santoro V. et al., Development of a High Intensity Neutron Source at the European Spallation Source: The HighNESS project, PREPRINT, arXiv:2204.04051. DOI: 10.48550/arXiv.2204.04051

Imaging and Energy Materials

ROOM

13:30-15:00

4D Neutron Imaging of Lithium Batteries and Fuel Cells

Ralf F. Ziesche[a], Nikolay Kardjilov[a], Tobias Arlt[b], Henning Markötter[c], Jennifer Hack[d], Paul R. Shearing[d,e], Dan J. L. Brett[d,e], Alessandro Tengattini[f,g], Cyrille Couture[f], Lukas Helfen[f], John Banhart[a,b] and Ingo Manke[a].

[a]*Helmholtz-Zentrum-Berlin, Berlin, Germany*
[b]Technische Universität Berlin, D-10623 Berlin, Germany
[c]*Bundesanstalt für Materialforschung und -prüfung, D-12205 Berlin, Germany* [d]Electrochemical Innovation Lab, UCL, London, UK
[e]The Faraday Institution, Didcot, UK
[f]Institut Laue-Langevin, Grenoble, France
[g]Univ. Grenoble Alpes, Grenoble, France

ralf.ziesche@helmholtz-berlin.de

Electrochemical storage systems, such as lithium batteries and fuel cells, have become an increasingly important pillar in a zero-carbon strategy for curbing climate change, with their potential to power multiscale stationary and mobile applications. Immense progress has been made in electrochemical storage technology during the past decades, but significant challenges remain, and new development strategies are required to improve performance, fully exploit power density capacity, utilize sustainable resources, and lower production costs. Suitable characterization techniques are crucial for understanding, inter alia, 3D diffusion processes, formation of passivation layers or dendrites in batteries or visualize the water management in fuel cells. Studies of such phenomena typically utilize 2D or 3D imaging techniques, offering locally resolved information. Over the last decades neutron imaging has been steadily growing in many disciplines as a result of improvements to neutron detectors and imaging facilities, providing significantly higher spatial and temporal resolutions. The high sensitivity for light-Z elements, in particular hydrogen and lithium, makes neutron imaging to the perfect probe to study inter alia, changes of the media distribution and transport mechanisms in electrochemical components.

With the instrument NeXT (Neutron and X-ray Tomograph), at ILL, a new high flux neutron imaging instrument joint the neutron community providing unique options for high spatial and temporal imaging ideal for multidimensional studies which is in the focus of that talk. After a short introduction about benefits of neutron imaging for the characterization of electrochemical devices, the focal point is on 4D (3D spatial and time resolved) imaging. Hereby, the advantages of dynamic, multi-dimensional and complementary imaging will be shown on examples. The main challenges for neutron imaging of electrochemical devices will be outlined and future developments of methods and their potential and significance for the electrochemical community will be discussed.

Investigation of silicon-based anodes for Li-ion batteries using X-ray and neutron 3D/4D imaging techniques

Erik Lübke[a], *Lukas Helfen*[a], *Roland Brunner*[b], *Thomas Vorauer*[b], *Jakub Drnec*[c], *Stefan Koller*[d], *Sandrine Lyonnard*[e]

[a]*Institut Laue-Langevin, Grenoble, France*
[b]*Materials Center Leoben Forschung GmbH, Leoben, Austria*
[c]*European Synchrotron Radiation Facility, Grenoble, France*
[d]*VATRA Innovation GmbH, Graz, Austria*
[e]*Univ. Grenoble Alpes – CEA – CNRS – IRIG-SyMMES, Grenoble, France*

lubke@ill.fr

Silicon-based anode materials are one of the most promising approaches to further increase the energy density of lithium-ion batteries. However, current materials are limited by poor cycling stability and rapid capacity fade, mainly caused by massive volume expansion of Si during lithiation and subsequent strain on the material composite. [1]

To optimize these materials, non-destructive techniques able to probe the local 3D morphology and Li content are necessary. By applying neutron computed tomography (NCT) in combination with X-Ray computed tomography (XCT), structural ageing and changes in the 3D distribution of lithium can be characterized for different charging states and recharge cycle numbers.

Industrially produced Li-ion battery coin cells containing a silicon-graphite composite anode material [2] were aged by performing 1, 300 and 700 charge-discharge cycles to reach remaining capacities of 100, 70 and 50 % respectively. NCT and XCT scans were acquired at the NeXT Instrument of the ILL in Grenoble [3]. These datasets were reconstructed, aligned using multi-modal registration and evaluated in specially developed data processing pipelines.

The 3D lithium distribution in the battery was determined at different cycle numbers and charge states by modelling the expected attenuation parameters to the observed values. From this information trapped Li can be identified and local changes in the distribution are analyzed to point out possible degradation and failure mechanisms.

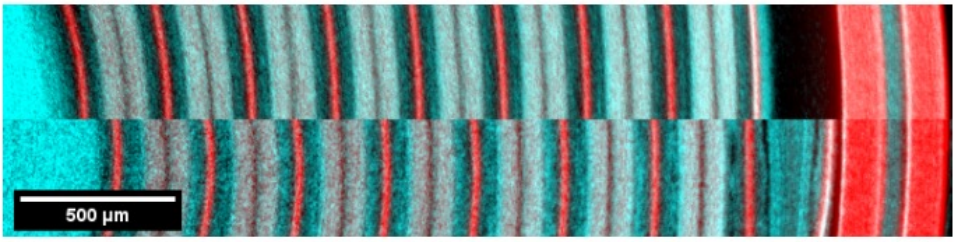


Figure 1. Cropped horizontal slices of Li-ion battery coin cells. Pixel size is 4.2 µm. Combined NCT (cyan) and XCT datasets (red). Comparison of an unaged reference cell (top) with a highly cycled cell (bottom).

We acknowledge financial support from the European Union's Horizon 2020 research and Innovation programs No. 875514 (ECO2LIB) as well as No. 847439 (InnovaXN) under the Marie Skłodowska-Curie grant agreement.

References

[1] T. Vorauer et al, https://doi.org/10.1038/s42004-020-00386-x
[2] C. Berhaut et al, https://doi.org/10.1021/acs- nano.9b05055
[3] A. Tengattini et al, https://doi.org/10.1016/j.ni- ma.2020.163939

Phase contrast neutron imaging of solid oxide electrochemical cells

E. B. Naver[a], P.K. Willendrup[b,c], R. Mokso[b], B. R. Sudireddy[a], P. Trtik[d], O. Yetik[d], M. Østergaard[e], H. Birkedal[e], S. Schmidt[c], L. Theil Kuhn[a].

[a]DTU Energy, Kgs. Lyngby, Denmark
[b]DTU Physics, Kgs. Lyngby, Denmark
[c]ESS DMSC, Copenhagen, Denmark
[d]LNS PSI, Villigen, Switzerland
[e]iNANO AU, Aarhus, Denmark

ebna@dtu.dk

Propagation-based phase contrast imaging is an established method in X-ray imaging. Our recent results suggest that a similar approach using a Paganin type filter [1] increases the neutron image contrast corresponding to a neutron flux increase of up to two orders of magnitude. We demonstrate here that by this method we can distinguish materials with low neutron absorption cross sections, which will increase temporal resolution of in-situ measurements. The neutron experiments were performed at the BOA beamline (SINQ@ PSI [2]); using the neutron microscope [3].

As proof-of-concept, we applied the method to a sample of thin Al and Zr alternating layers, each of low neutron absorption. We show that the layers are visible using propagation-based phase contrast imaging and compare this to a McStas [4] simulation. To simulate the phase contrast, a partial wave description of the neutron was added to McStas, by keeping track of how the neutrons change directions inside the sample and propagate them as waves after exiting the sample.

We applied the method to investigate the microstructure of two solid oxide electrochemical cell (SOC) samples. This microstructure is key to the performance in electrolysis or fuel mode, for gas to diffuse to the electrode where the reaction happens and for the particle surfaces that facilitate the reaction. The SOC samples consist of a dense electrolyte layer (Y2O3-doped ZrO2), a granular porous electrode layer (60% La0.4Sr0.4Fe0.03Ni0.03Ti0.94O3, 40% FeCr; or 60% Y2O3-doped ZrO2, 40% FeCr) and a porous metal support (FeCr), all with characteristic length scale 1-10 µm. The SOCs were also imaged by X-ray µCT to compare resolutions and contrast. The use of the Paganin-type filter increased the resolution and contrast between the FeCr particles and surrounding sample.

This demonstrates the potential for obtaining detailed structural information by propagation-based neutron phase contrast imaging. McStas is an invaluable tool to help assess the viability of the neutron phase contrast experiments.

We acknowledge support from the Danish ESS lighthouse on hard materials in 3D, SOLID.

References

[1] D. M. Paganin et al., arXiv, 1909.11186 (2019)
[2] M. Morgano et al., Nucl Instrum Methods Phys Res A 754, 46-56 (2014)
[3] P. Trtik et al., Physics Procedia 69, 169-176 (2015)
[4] P. K. Willendrup, and K. Lefmann, J Neutron Res 22, 1-16 (2020)

Magnetic bulk properties of ferromagnetic metal sheets

Alex Backs[a], Simon Sebold[b], Wai Tung Lee[c], Markus Strobl[d], Michael Schulz[b], Dmytro Orlov[a]

[a]Mechanical Engineering Sciences, Lund University, Lund, Sweden
[b]Heinz-Maier-Leibnitz Lentrum (MLZ), Technical University Munich, Garching, Germany
[c]Instrument Technologies Division, European Spallation Source (ESS), Lund, Sweden
[d]Paul Scherrer Institute (PSI), Villingen, Switzerland

alexander.backs@material.lth.se

Polarized neutron imaging (PNI) is a relatively new technique capable of probing the bulk magnetic properties and resolve spatial inhomogeneities in a material [1]. It requires a spin polarized neutron beam and relies on analysing the effect of a sample on the neutron spin, instead of the typical attenuation based approach of established imaging methods. In recent years, applications for PNI have been explored, including the imaging of large scale magnetic domains [2] or magnetic vector-fields [3].

Here, we present two case studies of magnetic domains in ferromagnetic metal sheets which use different modes of PNI, where the key advantage is the bulk sensitivity of PNI. This makes it an excellent complement to the established surface sensitive methods such as Kerr-effect microscopy [4]. In electrical engineering, ferromagnetic metals, especially silicon steels, are used as high performance magnetic core materials. Depending on the application, steels with diverse crystallographic and magnetic properties may be used.

In grain oriented (GO) silicon steel, crystal grains and magnetic domains are several millimetres large and the magnetization is preferentially uniaxial. Therefore, the neutron spin rotation in a sample is well defined and can be observed in a measurement, allowing an analysis of the magnetic domains properties and morphology. The technique reveals an unexpected discrepancy in the magnetization of domains with opposing magnetization directions.

In contrast, the grains and magnetic domains in non-grain oriented (NGO) nickel are in the low micrometre range. In this case, a statistical depolarization of a polarized neutron beam can be measured and evaluated. In thin sheets, indentures have been used to introduce local residual stress, which affects the magnetization process by restricting the magnetic domain wall mobility [4]. With PNI, the spatial extent of this effect can be visualized, which is currently investigated as a method to create magnetic flux barriers [5,6].

References

[1] M Strobl et al., Journal of Phys. D: Applied Physics 52 (2019) 123001
[2] K. Hiroi et al., Physica B: Condensed Matter 551 (2018) 146–151
[3] A. Hilger et al., Nature Communications, 9.1 (2018) 1-7
[4] H. Richert et al., Steel Research International 87.2 (2016) 232-240
[5] S. Vogt et al., Production Engineering 13.2 (2019) 211-217
[6] H. Weiss et al., Journal of Magnetism and Magnetic Materials 474 (2019) 643-653

Operando multimodal study of degradation and sodium storage processes in sodium-ion batteries

Domenico Battaglia[a], Anna Fedrigo[b], Daniel R. Sørensen[c], [a]Kristoffer V. Graae, Annabel Olgo[d], Alar Jänes[d], Enn Lust[d], [a]Salvatore de Angelis, Nikolaj Zangenberg[e], Søren Schmidt[f], Luise Theil Kuhn[a]

[a]DTU Energy, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark,
[b]ISIS Facility, UKRI-STFC, Rutherford Appleton Laboratory, Didcot OX11 0QX, United Kingdom,
[c]MAX IV Laboratory, Lund University, 224 84 Lund, Sweden,
[d]Institute of Chemistry, University of Tartu, 50411 Tartu, Estonia,
[e]Danish Technological Institute, Denmark, DK-8000 Aarhus C, Denmark,
[f]Data Management & Software Center, European Spallation Source, DK-2200 Copenhagen N, Denmark.

doba@dtu.dk

With neutron tomography and Bragg edge imaging, we can investigate sodium ion batteries (SIBs) in operando, to study the ionic transport, storage mechanisms (figure 1A) [1][2], and other phenomena such as degradation upon cycling and dendrite growth [3]. Throughout the project, we will investigate Prussian blue analogs and hard carbon, as promising electrode materials for SIBs, employing custom-built aluminum cells. Figure 1B shows a neutron radiography of such a cell containing a Prussian white cathode and a hard carbon anode. When the crystallographic properties of a material are dependent on the state of charge of the battery, being able to observe the crystallographic changes, makes possible to investigate the material's properties. During the project, we will complement the neutron data with X-ray techniques, such as micro-CT and X-ray diffraction. Recently, we performed an operando synchrotron diffraction experiment, where we could observe the crystallographic evolution of the electrodes at different charge states.

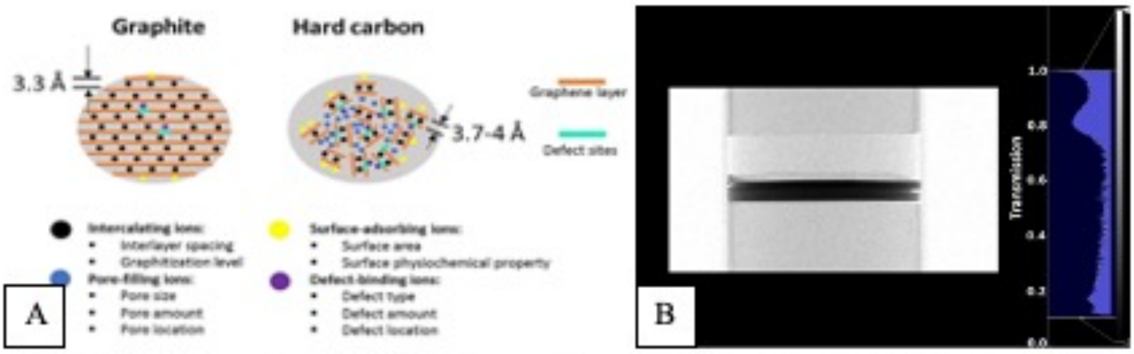


Figure 1. A) Storage mechanism of ions in graphite and hard carbon [2]. B) Neutron radiography of the custom-made cell. Layers from top to bottom: Prussian white, separator, hard carbon.

References
[1] M. J. Piernas Muñoz et al., Springer B. in Appl. Sci. and Tech., 2018, **2**, 9.
[2] Biwei Xiao, et al., ChemSusChem, 2019, **12**, 133
[3] Bing Sun, et al., Adv. Mater., 2020, **32**, 1903891

Large Scale Structures and the Life Sciences

ROOM

13:30-15:00

The myelin membrane visualised using photons, neutrons, and electrons

Petri Kursula[ab]

[a]*Department of Biomedicine, University of Bergen, Bergen, Norway,* [b]*Biocenter Oulu & Faculty of Biochemistry and Molecular Medicine, University of Oulu, Oulu, Finland*

petri.kursula@uib.no

Biological structures with self-assembling repetitive molecular assemblies are intriguing subjects for structural biology research. How do such biostructures form and function at the molecular level? My model system is myelin, a tightly packed multilayer important for the normal functioning of the vertebrate nervous system. Myelin consists of dozens of proteolipid membrane layers, wrapped by a glial cell around the axon, and myelin-specific proteins are directly involved in binding of myelin membranes together. These unique proteins have diverse structures but surprisingly similar functional properties. Our research focuses on the structure and properties of myelin-like membrane multilayers at different levels of detail. We use a combination of techniques, ranging from biophysics to structural biology [1-6], and experiments at large-scale photon and neutron infrastructures are crucial for the project. I will describe the molecular structure of the myelin membrane and discuss our recent results [1-6] and future plans with regard to more detailed studies on the molecular properties of myelin. Such information will be crucial for understanding both the molecular biology of myelin formation as well as mechanisms of demyelinating disease.

References

[1] Raasakka et al. (2017) Membrane association landscape of myelin basic protein portrays formation of the myelin major dense line. Sci. Rep. 7:4974.
[2] Raasakka et al. (2019) Molecular structure and function of myelin protein P0 in membrane stacking. Sci. Rep. 9:642.
[3] Laulumaa et al. (2019) A quasielastic neutron scattering investigation on the molecular self-dynamics of human myelin protein P2. J. Phys. Chem. B 123:8178-8185.
[4] Ruskamo et al. (2020) Cryo-EM, X-ray diffraction, and atomistic simulations reveal determinants for the formation of a supramolecular myelin-like proteolipid lattice. J. Biol. Chem. 295:8692-8705.
[5] Uusitalo et al. (2021) Human myelin protein P2: From crystallography to time-lapse membrane imaging and neuropathy-associated variants. FEBS J. 288:6716-6735.
[6] Ruskamo et al. (2022) Human myelin proteolipid protein structure and lipid bilayer stacking. Cell. Mol. Life Sci. 79: 419.

The role of hydrocarbons in cyanobacterial membranes

Sophie Ayscough[a], Melissa Sharp[a], Chi L. Chan[b], Andrew Curson[c], Oscar Ces[b], David Lea-Smith[c]

[a]European Spallation Source, Lund, Sweden
[b]Imperial College London, London, UK
[c]University of East Anglia, Norwich, UK

sophie.ayscough@ess.eu

Cyanobacteria contain highly-folded photosynthetic membranes called thylakoids, that host the necessary proteins for photosynthesis. It has been shown that these lipid membranes contain up to 17 mol% hydrocarbon in their compositions, which are synthesised by the bacteria.[1] Where the hydrocarbon sits within the lipid leaflets and the effect of hydrocarbon incorporation on thylakoid membrane properties has not previously been investigated. It is postulated that hydrocarbons facilitate curvature of the thylakoid membrane structures.

We have directly observed small angle scattering differences between two live bacterial samples: a wild-type cyanobacterium (WT) and a variant that has been genetically modified to not produce hydrocarbons. The thylakoids form stacked membranes in the cyanobacteria that scatter as Bragg peaks when measured by small angle scattering. Our data analysis suggests that there is a small decrease in thickness of membranes in the hydrocarbon deficient mutant. Furthermore, the hydrocarbon deficient mutant does not undergo the large light to dark membrane restructuring that would be expected, suggesting that the lack of hydrocarbons severely restricted the flexibility of the membrane.

To understand the underlying physics of alkane incorporation on lipid membrane properties we took the approach of investigating lipid bilayer membrane mimics. One of the key questions was to understand where the hydrocarbon sits within the lipid bilayer. We expect that the hydrocarbon will associate with the lipid tails, however, depending on the lipid properties, the alkane could sit parallel or perpendicular to the lipid tails within the bilayer leaflet. SANS results show an increase in bilayer thickness of a thylakoid lipid bilayer with increasing hydrocarbon content. By making use of selective deuteration (using deuterated hydrocarbon and hydrogenated lipid) we were able to observe that the hydrocarbon separates between the tail layers. A similar experiment was done on planar lipid bilayers with neutron reflectivity which further confirmed the formation of a hydrocarbon layer between the lipid leaflets.

One of the other approaches taken in this project has been determining the changes in dynamic properties of the lipid membranes with increasing hydrocarbon content. This has been investigated using infrared spectroscopy and differential scanning calorimetry of lipid films. Combined these initial results suggest that hydrocarbons increase the fluidity of the lipid bilayer-in line with our conclusions on live bacteria.

References

[1] Lea-Smith, D. J. et al. Hydrocarbons are essential for optimal cell size, division, and growth of Cyanobacteria. *Plant Physiol.* 172, 1928–1940 (2016).

Lipolysis of a thin triolein film

Ben Humphreys [a], José Campos Terán [b], Thomas Arnold [c], Philipp Gutfreund [d], Mario Campana [e] and Tommy Nylander [b]

[a] Physical Chemistry, Department of Chemistry, Lund University, Lund, Sweden
[b] Departamento de Procesos y Tecnología, Universidad Autónoma Metropolitana, CdMx, Mexico
[c] European Spallation Source – ESS, Lund, Sweden
[d] Institut Laue-Langevin – ILL, Grenoble, France
[e] ISIS facility, Rutherford Appleton Laboratory, Chilton, UK

ben.humphreys@fkem1.lu.se

The triglyceride aqueous interface has an important role in many processes, none more so than lipase activity. Lipases, such as *Thermomyces lanuginosus* lipase (TLL), are responsible for the degradation of triglycerides to smaller components, such as diolein, monoolein, glycerol and fatty acids. TLL attracts considerable interest in the food and pharmaceutical industries due to the catalytic behavior it possesses [1]. While the TLL lipolysis of triglyceride films has previously been investigated, [2,3] the influence of the reaction products on this process is poorly understood.

This aim of this study was to identify the chemical species present in a triolein film before and after TLL digestion and to monitor the physical properties of the film with high time resolution throughout the lipolysis after the addition of TLL. The thickness, mass, and elasticity of the film was continuously monitored over several hours enabling a detailed in-sight into the changes to the physical properties of the triolein film throughout these processes. [4] These findings were used to optimize the experimental design and sample preparation for the subsequent neutron reflectometry experiments performed at ILL and ISIS. Here, it was possible to measure the reflectivity profile of the equilibrated triolein film before and after exposure to lipase (Fig. 1) as well as monitor the kinetics of the lipolysis throughout the digestion.

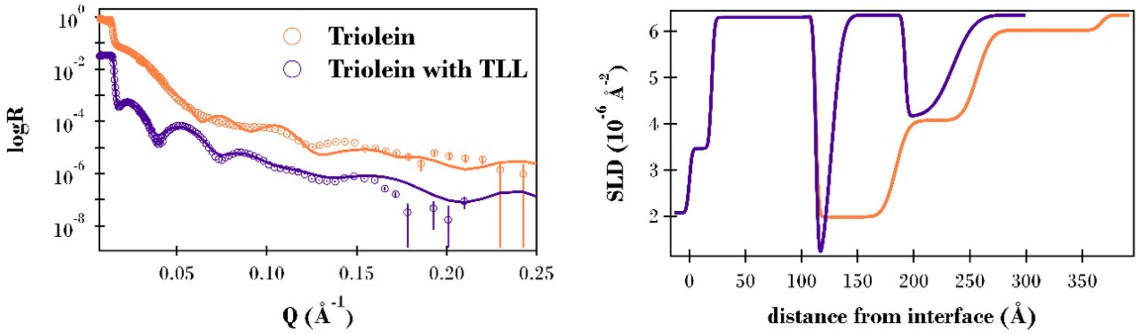


Figure 1. *Reflectivity measurements and SLD profiles for the triolein film before (orange) and after digestion (purple)*

References

[1] R. Fernandez-Lafuente, J. Mol. Catal. B Enzym., 2010, 62, 197
[2] A. Stamm et al. Chem. Phys. Lipids, 2018, 211, 37
[3] T. Snabe and S. Petersen, Chem. Phys. Lipids, 2003, 125, 69
[4] B. Humphreys et al. Front. Soft. Matter doi: 10.3389/frsfm.2022.929104

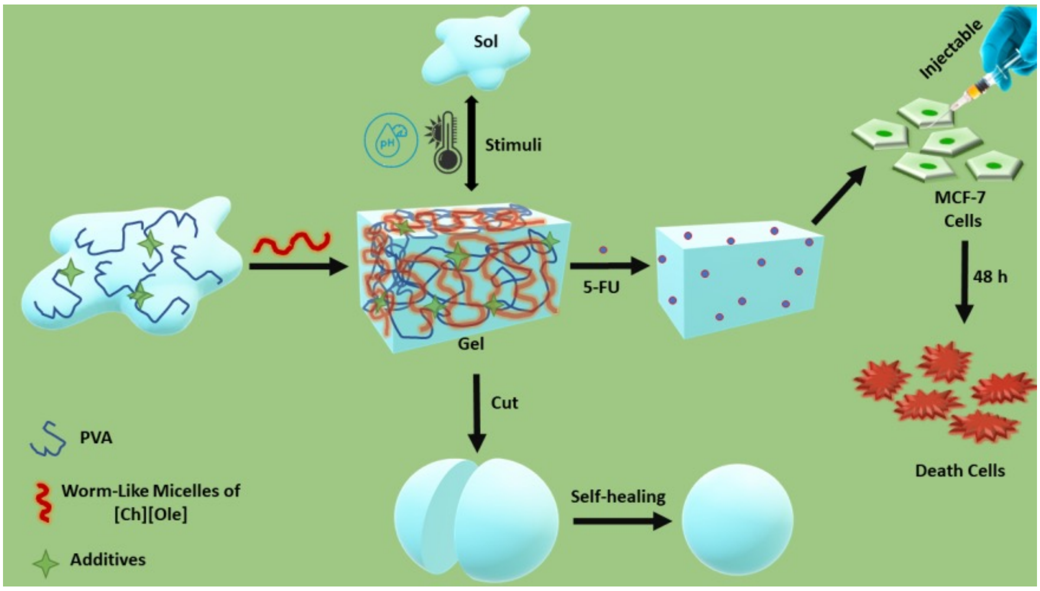
Multi responsive, self-healable and injectable ionic liquid based polymeric hydrogel for the treatment of Breast Cancer

Ravi Pansuriya[a], *Naved Malek*[a]

[a]*Ionic Liquids Research Laboratory, Department of Chemistry, Sardar Vallabhbhai National Institute of Technology, Surat-395007, Gujarat, India*

navedmalek@chem.svnit.ac.in

To ameliorate the limitations of traditional cancer therapies, scientists are working to produce safer, more effective and targeted drug carriers that increase its efficiency, reduce the negative effects of drug on the healthy cells, and aid in the prevention of cancer recurrences. In this paper, we developed self-healable, injectable, temperature and pH-responsive ionic-liquid based polymeric hydrogel as the local drug delivery system that exhibited sustained release of the antitumour drug, 5-Fluorouracil (5-FU). Poly (vinyl alcohol) and worm like micelle of choline oleate are cross linked with each other using borax as the crosslinker to form the hydrogel. Mechanical properties of the hydrogel were improved through the addition of glycerol and dextrose. Morphology, mechanical strength of hydrogel and interaction within the molecules are characterized via different analytical techniques, including ATR-FTIR, small-angle neutron scattering (SANS), SEM and rheological measurement. The in vitro cytotoxicity (MCF-7) and in vitro drug release study indicated that hydrogel can be used as the targeted delivery of 5-FU at physiological condition and more effectivity killing the cancerous cells. Overall, the study shows the effectiveness of the hydrogel system by determining that whether hydrogel formulation supported or hampered total cellular 5-FU trafficking.



A clear picture of the active site of hen egg-white lysozyme from atomic resolution neutron crystallography

Joao Ramos[a,b,c], Valerie Laux[b], Kay Diederichs[d], Michael Haertlein[b], V. Trevor Forsyth[b,e,f], Estelle Mossou[g], Sine Larsen[h], Annette E. Langkilde[c]

[a]Department of Molecular Biology and Genetics, Aarhus University, Universitetsbyen 81, 8000 Aarhus C, Denmark
[b]Institut Laue-Langevin, 71 avenue des Martyrs, 38000 Grenoble, France
[c]Department of Drug Design and Pharmacology, University of Copenhagen, Universitetsparken 2, DK-2100 Copenhagen, Denmark
[d]Department of Biology, University of Konstanz, Box 647, D-78457 Konstanz, Germany [e]Faculty of Medicine, Lund University, 221 00 Lund, Sweden
[f]LINXS Institute for Advanced Neutron and X-ray Science, Scheelvagen 19, 223 70 Lund, Sweden
[g]European Synchrotron Radiation Facility, 71 avenue des Martyrs, 38000 Grenoble, France.
[h]Department of Chemistry, University of Copenhagen, Universitetsparken 5, DK-2100 Copenhagen, Denmark

ramosj@mbg.au.dk

Neutron crystallography has played a crucial role in deciphering enzymatic mechanisms by determining the positions of hydrogen/deuterium atoms in proteins [1-2]. H atoms constitute approximately 50% of all protein atoms and are crucial to protein structure and dynamics, and to the interactions with substrates and ligands [3]. On the other hand, H atoms remain elusive to X-ray crystallography, even when atomic resolution is available. To date, the highest resolution neutron structure deposited in the Protein Data Bank is that of hydrogenated crambin – buffer exchanged to D_2O – at 1.1 Å resolution and 78% complete [4]. Due to the presence of H atoms in non-exchangeable positions and to the lack of diffraction data completeness, the full potential of atomic resolution neutron crystallography is yet to be demonstrated.

We have obtained the crystal structures of hen egg-white lysozyme in its hydrogenated (H-HEWL) and perdeuterated (D-HEWL) variants from complete neutron diffraction data at 1.1 Å and 0.91 Å resolution, respectively. These neutron structures provide a detailed picture of lysozyme's active site, specifically of the configuration of important residues (*e.g.*, Asn44 and Asn46) and water molecules, which are often misinterpreted in X-ray crystallography studies. Comparative analysis between the two datasets reveals that protein perdeuteration results in significant benefits to neutron data quality and to the accuracy of the resulting model. In contrast, neutron maps from the hydrogenated sample seem to preclude relevant structural detail, *e.g.*, protein residue disorder.

References

[1] Cuypers, M. G., et al. (2013). Angew Chem Int Ed Engl **52**(3): 1022-1025.
[2] Gajdos, L., et al. (2022). Nat Commun **13**(1): 194.
[3] Gajdos, L., et al. (2021). Structure **29**(9): 1003-1013 e1004.

Spectroscopy and Magnetism

ROOM

13:30-15:00

Topological magnon band structure of emergent Landau levels in a skyrmion lattice

*T. Weber[a], D. Fobes[b], J. Waizner[c], P. Steffens[a], G. S. Tucker[d], M. Böhm[a],
L. Beddrich[e], C. Franz[e], H. Gabold[e], R. Bewley[f], D. Voneshen[f], M. Skoulatos[e], R. Georgii[e],
G. Ehlers[g], A. Bauer[e], C. Pfleiderer[e], P. Böni[e], M. Janoschek[h], and M. Garst[i]*

[a]Institut Laue-Langevin, Grenoble, France
[b]Los Alamos National Laboratory, USA
[c]University of Cologne, Germany
[d]European Spallation Source ERIC, Lund, Sweden
[e]Technical University of Munich, Germany
[f]ISIS Facility, Didcot, UK
[g]Oak Ridge National Laboratory, Oak Ridge, USA
[h]Paul-Scherrer-Institut, Villigen, Switzerland
[i]Karlsruhe Institute of Technology, Germany

tweber@ill.fr

Research into the two-dimensional, vortex-like skyrmion order has gained much experimental and theoretical interest since its discovery over a decade ago [1], where a skyrmion lattice could be identified in the itinerant magnet MnSi at temperatures just below 30 K and magnetic fields around 0.2 T [1]. Here, we present the first comprehensive study of magnon dynamics in the skyrmion phase of MnSi [2]. In first order, magnetic dynamics is governed by the exchange and the Dzyaloshinskii-Moriya interaction terms, where the latter has profound consequences for all magnetic phases. Namely, it causes magnon creation and annihilation to take place at two distinct energy levels. This phenomenon is restricted to reduced momentum transfers having a component perpendicular to the two-dimensional skyrmion plane. The dynamical magnetic structure factor becomes asymmetric ("non-reciprocal"). Apart from the skyrmion phase, such an asymmetric behaviour could also be observed in the topologically trivial phases of MnSi [3, 4, 5].

To map out the magnetic dynamics in the skyrmion phase of MnSi we employed polarised triple-axis, time-of-flight, and spin-echo techniques [2]. Apart from an in-depth analysis of the non-reciprocal dynamics, we succeeded in observing a splitting of the magnon energies into closely-spaced Landau levels for reduced momentum transfers inside the skyrmion plane. Theoretically, we describe our results using a mean-field linear spin-wave model which is phenomenologically inspired by the band structure theory of electrons. A correction of the theoretical model for instrumental resolution yields an excellent quantitative agreement between experiment and theory.

References

[1] S. Mühlbauer *et al.*, Science 323 (5916), pp. 915–919 (2009).
[2] T. Weber *et al.*, Science 375 (6584), pp. 1025–1030 (2022).
[3] T. J. Sato *et al.*, Phys. Rev. B 94, 144420 (2016).
[4] B. Roessli *et al.*, Phys. Rev. Lett. 88, 237204 (2002).
[5] T. Weber *et al.*, Phys. Rev. B 97, 224403 (2018); T. Weber *et al.*, Phys. Rev. B 100, 060404(R) (2019).

High-resolution spectrum of fractional excitations in $Ce_2Sn_2O_7$

Victor Porée[a], Elsa Lhotel[b], Sylvain Petit[c], Han Yan[d], Markus Appel[e], Jacques Ollivier[e], Tom Fennell[a], Andriy Nevidomskyy[d] & Romain Sibille[a]

[a]Paul Scherrer Institut, Switzerland
[b]Institut Néel, CNRS, France
[c]CEA Saclay, LLB, France
[d]Rice University, USA
[e]Institut Laue-Langevin, Grenoble, France

romain.sibille@psi.ch

A correlated liquid state was reported in the pyrochlore $Ce_2Sn_2O_7$ [1]. Its nature remained elusive, but with additional knowledge on the crystal-electric field scheme, the case of cerium pyrochlores was further investigated based on degrees of freedom having both magnetic dipole and magnetic octupole components [2-5]. A number of recent studies [2-7] all point towards a quantum spin ice (QSI) based on a manifold of ice-rule correlated octupoles to be stabilized in these materials. Theorists had conceptualized such an octupolar QSI [8-9], where quantum dynamics is endowed by couplings between other components of the 'dipole-octupole' pseudo-spins.

This talk reviews findings reported so far on $Ce_2Sn_2O_7$ [1-2], puts these in perspective with results on other cerium pyrochlores [3-7], and presents new experimental data that further hint at cerium pyrochlores being genuine representatives of QSI – the model 3D quantum spin liquid. In particular, we argue that excitations observed in Time-Of-Flight spectroscopy are associated with the fractional excitations of QSI. We further confirm this conclusion by presenting results of backscattering experiments performed on IN16B. The energy resolution improved by more than an order of magnitude compared to previous works [2-4] allows detailed comparisons with theories for spinon dynamics in QSI. It is predicted that the alternative vacuum of this condensed-matter system is drastically different from that of our Universe, with phenomena arising from strong light-matter interactions (10-11). We observe a gap spectrum with an edge-structure corroborating the predictions for the effects of photons on the production of matter excitations.

References

[1] Sibille et al., Phys. Rev. Lett. 115 097202 (2015)

[2] Sibille et al., Nature Phys. 16, 546-552 (2020)

[3] Gaudet et al., Phys. Rev. Lett. 122 187201 (2019)

[4] Gao et al., Nature Phys. 15, 1052-1057 (2019)

[5] Porée et al., Phys. Rev. Materials 6, 044406 (2022)

[6] Bhardwaj et al., npj Quantum Mater. 7, 51 (2022)

[7] Smith et al., Phys. Rev. X 12, 021015 (2022)

[8] Huang, Chen & Hermele, Phys. Rev. Lett. 112, 167203 (2014)

[9] Li & Chen, Phys. Rev. B 95, 041106 (2017)

[9] Udagawa & Moessner, Phys. Rev. Lett. 122, 117201 (2019)

[10] Morampudi, Wilczek & Laumann, Phys. Rev. Lett. 124, 097204 (2020)

[11] Pace, Morampudi, Moessner & Laumann, Phys. Rev. Lett. 127, 117205 (2021)

Singlet magnetism in $SrTm_2O_4$

Abhijit Bhat Kademane[a], *Diana Lucia Quintero Castro*[a], *Rasmus Toft-Petersen*[b,c]

[a] *Department of Mathematics and Physics University of Stavanger, Norway*
[b] *Department of Physics, Technical University of Denmark, Denmark*
[c] *European Spallation Source, Lund, Sweden*

abhijit.bhatkademane@gmail.com

The $SrTm_2O_4$ belongs to the family of orthorhombic rare-earth oxides where two inequivalent rare-earth sites form zig-zag chains along the c-axis and distorted honeycomb in the ab-projection. The previous report on this system has shown that the system does not order down to 65mK [1]. Here we present various competing interactions and their relative strengths to understand the lack of an ordered ground state.

Diffused scattering measurements with *XYZ*-polarization (D7@ILL) show a broad peak centered at 1.3 $Å^{-1}$. However, this diffused signal originates from the excited states, thus confirming the lack of short-range order in the system.

To explain the single-ion effects, two unique crystal field models were implemented; DFT based method that uses Wannier functions and the Effective charge (EC) model. The EC model predicts a singlet ground state for both Tm sites [2]. Two-ion behavior was studied by modeling the dispersing excitations using RPA with only exchange interactions. The RPA results suggest that the Tm1 chain is in dimer limit, whereas Tm2 chains are frustrated. The critical ratio estimated under RPA shows crystal fields to be two orders of magnitude stronger than the exchange interactions, and it is impossible to drive this system to a thermal second-order phase transition.

Application of magnetic field greater than 4T induces ordering. The NPD results with magnetic symmetry analysis reveal that the Tm2 chains are polarized in the applied field while the dimer chains (Tm1 chains) start ordering into the XY-AFM phase. Additionally, unique zero-field μSR results show signs of long-range order contradicting Ref. [1,2]. However, we show that the muon implantation distorts the crystal fields and leads to nuclear hyperfine enhancement and thus concurring with other results.

References

[1] H. Li et al. J. Mater. Chem. C, 3, 7658-7668 (2015).
[2] A. B Kademane et al. JMMM, 551, 169020 (2022).

AI-assisted neutron spectroscopy – Log-Gaussian processes for TAS

A. Schneidewind[a], M. Teixeira Parente[a], G. Brandl[a], C. Franz[a], U. Stuhr[b], M. Ganeva[a]

[a]Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich, Garching, Germany
[b]Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institute (PSI), Villigen, Switzerland

a.schneidewind@fz-juelich.de

Three-axes spectroscopy (TAS) is a well-established method that has not substantially changed in the past decades of its use. Nowadays, with increasing request and limited availability of TAS instruments, application of AI methods is one option to increase the efficiency of TAS.

From the perspective of an AI method, TAS experiments collect noisy observations of a 2D intensity function to investigate a material of interest. If the material's intensity structure is unknown, experimenters usually decide manually where to place measurements for a rapid overview. AI methods can assist this process by choosing informative measurement locations while taking instrument costs into account and hence optimize the available beam time in this mode. For example, avoiding measurements in the background (no signal) but preferring regions of signal leads to more efficient measurements. Our method [1] for discovering regions of signal is based on Gaussian Process Regression as a technique for probabilistic function approximation and makes use of log-normal distributions. Our approach focuses on implementing instrument parameters into the cost function (e.g., different moving times for energy- / Q-scans), managing noise and background functions, and respects weak as well as strong intensities to avoid loss of information. For simple dispersions like intensity-modulated phonons (see Fig. 1), the full information can be achieved instead of an overview only within reasonably short amount of experimental time.

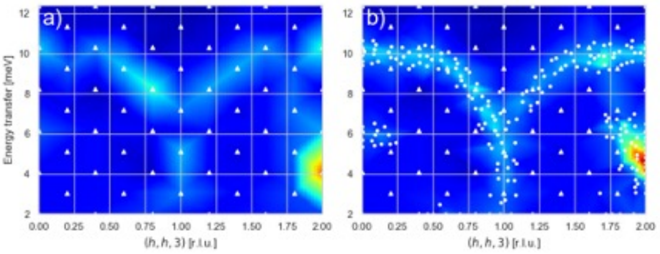


Fig. 1: AI-assisted measurement of an intensity-modulated, coupled phonon on EIGER/PSI. a) Initialization (~3h). b) Result (~10.5h).

The algorithm was tested on simulated data for several typical functions (CEF, phonons, SDW, dispersion of frustrated systems) and experimentally on EIGER/PSI. In order to quantify the benefit of our approach, we present results of a benchmarking procedure [2] that we have developed as a cost-benefit analysis in a synthetic but still representative setting.

References:

[1] Teixeira Parente M. et al. M., arXiv e-prints, 2105.07716 (2021).
[2] Teixeira Parente M. et al., Front. Mater. **8**, 772014 (2022).

Magnetic structure and spin waves of the doped cobalt oxide $La_{2-x}Ba_xCoO_4$

Siobhan M Tobin[a], Dharmalingam Prabhakaran[a], Helen Walker[b], Ketty Beauvois[c], Bachir Ouladdiaf[c], Andrew Boothroyd[a].

[a]*University of Oxford, Oxford, United Kingdom*
[b]*ISIS Neutron and Muon Source, Didcot, United Kingdom*
[c]*Institut Laue-Langevin, Grenoble, France*

siobhan.tobin@physics.ox.ac.uk

The role of charge stripes in cuprate superconductors is not yet understood. The undoped cuprates are antiferromagnetic Mott insulators. A simple model is that doping disrupts the AFM order and the introduced holes form stripes, with the periodicity and values of charge stripes affected by dopant concentration x [1,2]. There is competition between different forms of charge and magnetic order, manifesting in tension between the pseudogap and superconducting phases. Insights into the spin and charge stripes may be gleaned from studying isostructural compounds that share the same parent phase as the cuprates, but remain insulating at low temperatures and over a wide range of dopant concentrations, such as the cobalt oxides [3]. Stripes in superconducting $La_{2-x}Ba_xCuO_4$ have been shown to be more stable than those found in $La_{2-x}Sr_xCuO_4$; hence, we have investigated $La_{2-x}Ba_xCoO_4$ as a candidate striped insulator. We studied crystals of $La_{2-x}Ba_xCoO_4$ at two dopant concentrations $x = 1/2$ and $x = 1/3$ using neutron scattering. We will present single crystal diffraction data from ILL D10 highlighting magnetic and possible charge order. Neutron time-of-flight data from the ISIS MERLIN instrument displays strong spin wave dispersion and we will compare this to the characteristic hour-glass dispersion of other cobalt oxides [3]. Finally, we will discuss the connection between charge stripes and hour-glass spin wave spectra.

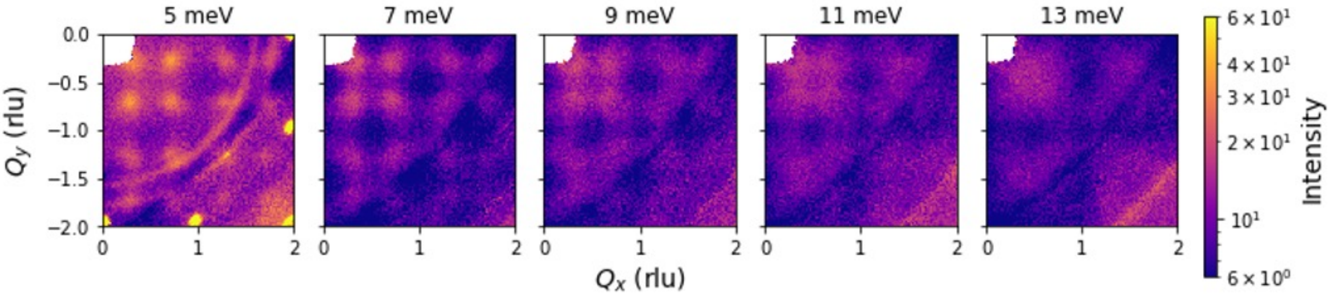


Figure: Time-of-flight data showing the low energy spin wave dispersion of $La_{1.5}Ba_{0.5}CoO_4$.

References

[1] Tranquada, J.M., *Adv. Phys.* **69** 437 (2020)
[2] Emery, V.J., Kivelson, S.A., and Tranquada, J.M., *PNAS* **96** 8814 (1999)
[3] Boothroyd, A.T. et al., *Nature* **471** 341 (2011)

Neutrons and Current Climate Challenges

Stora Salen

09:00-10:30

Complex hydrides for energy applications

Wiebke Lohstroh[a]

[a]Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Germany

wiebke.lohstroh@frm2.tum.de

The current climate challenges, the associated unalienable need for a sustainable energy economy and eventually net-zero CO_2 emissions require comprehensive efforts in all areas of the energy sector to improve efficiency and the minimize CO_2 release. In the area of materials science, research with neutrons provides unique tools to address pertinent questions for the further development of efficient, cost effective and reliable energy materials. Example include the characterization of high performance superalloys for higher energy efficiency, studies on energy conversion and aging phenomena in established and novel battery systems, fuels cells or solar cells, as well as the investigation solid state hydrogen storage materials.

In this presentation, we will give an overview on recent results with a focus on light weight solid state complex hydrides. These materials are salt like compounds of general composition $A[TH_x]_n$ where A is an alkaline or alkaline earth ion (Li, Na, Mg) and T= Al, B or N. For some compounds of this materials class, an unusual high ionic conductivity of the Li or Mg ions has been observed which is strongly correlated with the mobility the hydrogen entities [1], and as such, the materials are both studies for application in reactive hydride composites for hydrogen storage applications as well as for use in all solid state battery applications.

References

[1] Lohstroh, Wiebke, and Michael Heere, J. Phys. Soc. of Japan 89 (2020): 051011.

Neutron scattering studies on CO_2 confined in nanoporous materials: Applications to CO_2 sequestration and oil recovery

Konstantinos L. Stefanopoulos

NCSR "Demokritos", Institute of Nanoscience & Nanotechnology, Athens, Greece

k.stefanopoulos@inn.demokritos.gr

Fluids when confined within nanopores have unique thermodynamic, structural and dynamic properties that vary significantly from those in the bulk phase because of the finite pore size and the fluid interaction with the pore wall. Total neutron scattering combined with small-angle neutron scattering (SANS) can be proved to be powerful tools for the investigation of the location, the structure, and the phase behaviour of fluids confined within the pores [1,2].

We have carried out a series of neutron experiments on CO_2 confined within pores with well-defined geometry at various thermodynamic states mainly at ISIS Neutron and Muon Source, STFC Rutherford Appleton Laboratory. We have mapped the structural properties of sorbed CO_2 at sub- and supercritical states as well as upon freezing for these model pore systems such as ordered mesoporous silicas (MCM-41, SBA-15) and carbons (CMK-3) [3].

Having gained the knowledge of CO_2 confined in well-defined pore systems we have further utilised *in situ* CO_2 sorption and neutron scattering techniques on complex pore networks in order to explore the pore morphology as well as the pore accessibility and the structural properties of confined CO_2. This information can be important in the case of sedimentary rocks for the design of optimal CO_2 sequestration as well as gas and oil recovery projects. It is noteworthy that carbon capture and storage (CCS) has been recognized key technology to reduce greenhouse gas emissions in an attempt to limit the effects of global climate change. The potential of combining CO_2-enhanced oil recovery (EOR) technology with CCS has the benefits of EOR and permanent storage of an amount of injected CO_2 in the depleted reservoir. In case of Marcellus Shale, the neutron scattering results indicated that a large fraction of pores was inaccessible to CO_2, suggesting that despite the vast numbers of micropores in shale, most of these pores would be unavailable for storage for geologic CO_2 sequestration [4]. Finally, in a recent neutron experiment we monitored *in situ* decane displacement by injecting supercritical CO_2 on a limestone filled with decane [5]. Again, the results suggested that small mesopores have low accessibility to CO_2.

References

[1] Alba-Simionesco C, Coasne B, Dosseh G, et al., J. Phys. Condens. Matter 18 (2006) R15.
[2] Melnichenko YB, Small-Angle Scattering from Confined and Interfacial Fluids: Applications to Energy Storage and Environmental Science, Springer (2015).
[3] Steriotis TA, Stefanopoulos KL, Katsaros FK, et al., Phys. Rev. B 78 (2008) 115424; Stefanopoulos KL, Katsaros FK, Steriotis TA, et al., Phys. Rev. Lett. 116 (2016) 025502; Stefanopoulos KL, Tampaxis C, Sapalidis AA, et al., Carbon 167 (2020) 296.
[4] Stefanopoulos KL, Youngs TGA, Sakurovs R, et al., Environ. Sci. Techn. 51 (2017) 6515.
[5] Stefanopoulos KL, Favvas EP, Karanikolos GN, et al., Energy Adv. 1 (2022) 67.

In situ flow exchange experiments between macropore and soil matrix combining neutron and X-ray tomographies

Luis Alfredo Pires Barbosa[a] *and Horst H. Gerke*[a]

[a]*Research Area 1 "Landscape Functioning", Working Group "Hydropedology", Leibniz Centre for Agricultural Landscape Research (ZALF), Eberswalder Strasse 84, 15374 Müncheberg, Germany*

luispiresbarbosa@zalf.de

Preferential pathways (i.e. macropores) may be formed as a result of biotic (e.g. earthworm burrows, decayed root channels) or abiotic process (i.e., cracks caused by wetting-drying forces). The extent to which water infiltration bypasses the soil matrix affects root zone drainage and plant nutrient transport and the filtering function of soil by potentially increasing leaching of chemicals to groundwater [1]. By combining synchrotron-based neutron and X-ray tomographies [2], the key challenge of explaining the dynamics of soil structure and preferential flow may be accomplished [3]. Therefore, flow exchange experiments were carried out in Neutron and X-ray beam lines (Fig. 1a) of intact soil samples with earthworm burrows coated with organic matter (Fig. 1b). The biopore-matrix mass exchange was quantified during preferential flow imposed by applying a water film on the biopore surface using 4D (3D plus time) X-ray and Neutron tomography imaging. From image analysis (Fig. 1), flow exchange between biopore and matrix was greater for regions without organic coating.

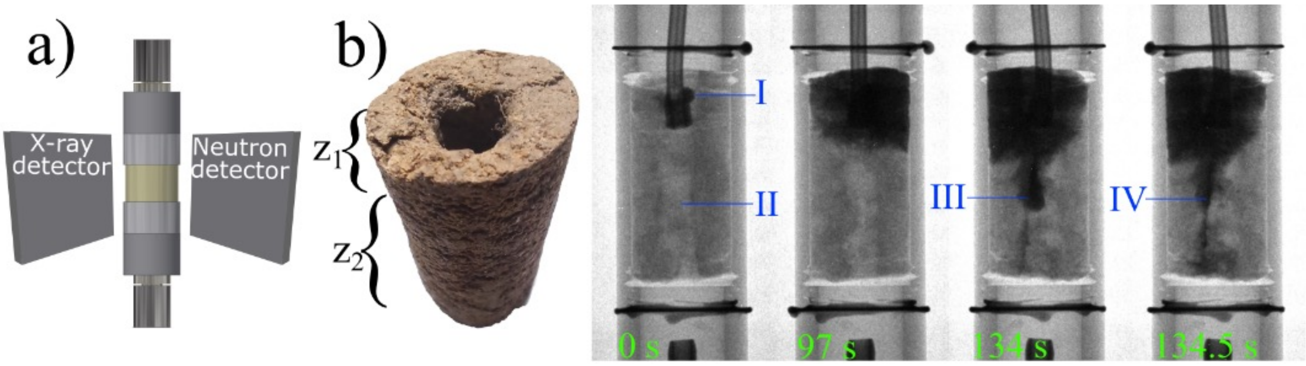


Fig. 1. Flow exchange experiments. Z1 and Z2 are regions without and with visible coating. Injection of water (I) in the biopore surface at initial instant (t=0s). Saturation of the top layer (t=97s). Formation of water film in the bottom part of the biopore surface (III) and propagation of the water film along the biopore length (IV).G

References

[1] J. M. Köhne, S. Köhne, and J. Šimůnek, 'A review of model applications for structured soils: a) Water flow and tracer transport', *J. Contam. Hydrol.*, vol. 104, no. 1–4, pp. 4–35, 2009, doi: 10.1016/j.jconhyd.2008.10.002.

[2] A. Tengattini *et al.*, 'NeXT-Grenoble, the Neutron and X-ray tomograph in Grenoble', *Nucl. Instruments Methods Phys. Res. Sect. A Accel. Spectrometers, Detect. Assoc. Equip.*, vol. 968, no. October 2019, p. 163939, 2020, doi: 10.1016/j.nima.2020.163939.

[3] H. Vereecken *et al.*, 'Modeling Soil Processes: Review, Key Challenges, and New Perspectives', *Vadose Zo. J.*, vol. 15, no. 5, p. vzj2015.09.0131, 2016, doi: 10.2136/vzj2015.09.0131.

Investigating the physical behaviour of novel cyclic poly(lactone)s using SANS

Philip B. Yang[a], Matthew G. Davidson[a], Karen J. Edler[a], Steven Brown[b]

[a]Bath, United Kingdom
[b]Wollaston, United Kingdom

py300@bath.ac.uk

Cyclic polymers have long been reported in literature, but their development has been stunted by synthetic issues and the presence of linear contaminants. In the past 15 years there have been many advances in synthetic methods which have allowed pure cyclic polymers to be synthesized with relative ease. The absence of end groups in these polymers provides interesting physical properties and raises important questions regarding their rheology which have yet to be fully answered, such as the way in which they relax stress. The physical behaviour of cyclic polymers needs to be understood in order to properly harness their potential.1

A key aim of our work has been the synthesis of novel bio-based cyclic polymers, with the goal to use the varied physical properties of the cyclic topology to improve the commercial viability of bio-based polymers. We have synthesized cyclic poly(lactide) (cPLA) and cyclic poly(ε- decalactone) (cPDL) and confirmed their pure cyclic nature using GPC, MALDI-TOF, DSC and NMR results. Through polymerization of l-lactide and racemic lactide, we have synthesized cPLLA, atactic cPLA and highly heterotactic cPLA (Pr = 0.77). Such tacticity control of cPLA has yet to be reported to our knowledge. cPDL has yet to be reported in literature and is a novel bio-based cyclic polymer.

Using SANS at ILL, we investigated the physical behaviour of these polymers in comparison to each other and to comparable linear PLA/PDL samples. The polymers were analysed at two tem- peratures (15 and 40 C) in two deuterated solvents (acetone-d6 and THF-d8) over a wide q range (0.003 to 0.52 A-1). The data was then fitted to the RPA model in SASview for comparisons of effective polymer-solvent interaction parameters and the Flory exponent. This type of study has only been reported using limited poly(styrene) samples and the variation of monomer choice and tacticity at varying molecular weights and temperatures has allowed for new comparisons and exciting results which may shed light on the physical intricacies of the cyclic topology.2 These results included substantial differences between the different tacticities of cPLA specifically and a much greater influence of temperature on cPLA results compared to previous literature.

References

[1] Yang, P. B.; Davidson, M. G.; Edler, K. J.; Brown, S. Synthesis, Properties, and Applications of Bio-Based Cyclic Aliphatic Polyesters. Biomacromolecules 2021. https://doi.org/10.1021/acs.biomac.1c00638.

[2] Gartner, T. E.; Haque, F. M.; Gomi, A. M.; Grayson, S. M.; Hore, M. J. A.; Jayaraman, A. Scaling Exponent and Effective Interactions in Linear and Cyclic Polymer Solutions: Theory, Simulations, and Experiments. Macromolecules 2019. https://doi.org/10.1021/acs.macromol.9b00600.

The magnetic flux line – a tool for exploring unconventional superconductors

Elizabeth Blackburn[a]

[a]Division of Synchrotron Radiation Research, Lund University, SE-22100 Lund, Sweden

elizabeth.blackburn@sljus.lu.se

Over thirty years ago, the high-temperature cuprate superconductors were discovered, leading to a flurry of work that established that they did not obey the strictures of the Bardeen-Cooper-Schrieffer (BCS) theory for superconductors, although many of the phenomenological tools that had been developed prior to this could still be applied. It became clear that a Cooper pair of electrons formed, but that the superconducting energy gap had become more complicated. At the same time, other families of so-called 'unconventional' superconducting states were being discovered, creating a complicated landscape where quantitative predictions have proved elusive. Many varied experimental techniques have been deployed on these materials. Taking the cuprate superconductors as an example, it is now clear that the copper-oxygen planes that are thought to drive the superconductivity are prone to a wide range of electronic instabilities that evolve as a function of the doping.

Most of these unconventional superconductors fall into the class known as Type-II, meaning that above a certain critical field, magnetic field can penetrate deep into the material, but only in the form of lines of magnetic flux, creating magnetic vortices. This creates a network of normal (non-superconducting) state in the vortex core. These vortices are very sensitive to the nature of the superconductivity and can tell us about properties like the London penetration depth (how far magnetic field extends into the pure superconducting state) and the coherence length (roughly the size of the Cooper pair). Using neutron diffraction, we can explore how these properties vary throughout the superconducting phase of a material. In some cases, we observe unexpected deviations, and in this talk I will explore what we can draw from this [1-3].

References

[1] E. Campillo *et al.*, Phys. Rev. B 104, 184508 (2021).
[2] E. Campillo *et al.*, Phys. Rev. B 105, 184508 (2022).
[3] A. Cameron & E. Campillo *et al.*, arXiv:2208:06706 (2022).

Gas hydrates: from their natural environment to energy innovation

Arnuad Desmedt

Institut des Science Moléculaire, Groupe de Spectroscopie Moléculaire, Université de Bordeaux I
351 Cours de la Libération, F-33405 TALENCE Cedex

Arnaud.desmedt@u-Bordeaux.fr

Abstract Text

References

Development of an Ultra Cold and Very Cold Neutron Source at the European Spallation Source

Valentina Santoro

On behalf of the HighNESS consortium

European Spallation Source ERIC, P.O. Box 176, SE-221 00, Lund, Sweden

valentina.santoro@ess.eu

The HighNESS project, a design study supported by 3MEuro Research and Innovation Action within the EU Horizon 2020 program, is now underway to develop a second neutron source below the spallation target. Compared to the first source, located above the spallation target and designed for high cold and thermal brightness, the new source will provide higher intensity, and a shift to longer wavelengths in the spectral regions of Cold, Very Cold (10-120 Å wavelength range), and Ultra Cold neutrons UCNs (>500 Å). Concerning the design of the Ultra Cold Neutron and Very cold neutron source, a digital workshop has been held from February 2nd to February 4th, 2022 where experts from various laboratories and Universities have gathered to propose and discuss ideas and challenges for the development of these sources. During the course of the workshop, several possibilities have been identified on where to locate the VCN and UCN sources. The UCN source could be placed in close vicinity or at some distance from the primary cold source as shown in Figure 1. Regarding the VCN source, we have identified two possibilities. In the first option, the VCNs are extracted from the main CN source using advanced reflectors [1]. While in the other case we make use of a dedicated VCN converter, for which a material capable of delivering a high flux of VCNs is needed. From the point of view of neutronic performance, two promising materials, which are under study in the HighNESS project, are solid deuterium at about 5 K and deuterated clathrate hydrates around 2 K. In the talk I will discuss all these possibilities that are currently under study as a part of the HighNESS project.

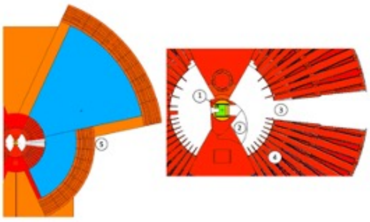


Figure 1: The possible locations of UCN sources, as studied within the HighNESS project are: (1) inside the "twister", a structure which contains the upper (liquid-parahydrogen, high-brightness) and lower (liquid-deuterium, high-intensity moderators; (2) inside the moderator cooling block, (3) in the large beamport (shown as a white segment in the monolith) that is initially foreseen to be used for the NNBAR experiment, (4) in a standard beamport, (5) outside the "bunker", a heavy concrete shielding structure (shown in orange) placed around the monolith.

References

[1] L. Zanini et all, “Very Cold and Ultra Cold Neutron Sources for ESS” proceeding of the Workshop of Very Cold and Ultra Cold Neutron Sources for ESS”

Molecular insights into protein - membrane interactions: endocytosis, fusion and neutrons

Nathan R. Zaccai

University of Cambridge, Cambridge, United Kingdom

nrz20@cam.ac.uk

Coronavirus disease-2019 (COVID-19), a potentially lethal respiratory illness caused by the coronavirus SARS-CoV-2, emerged in the end of 2019 and has since spread aggressively across the globe. A thorough understanding of the molecular mechanisms of cellular infection by coronaviruses is therefore of utmost importance. A critical stage in infection is the fusion between viral and host membranes, as the virus enters the host cell.

We have recreated important elements of the membrane fusion mechanism of the SARS-CoV-2 coronavirus by simplifying the system down to its core elements, amenable to experimental analysis by neutron scattering. Neutrons are well suited for the study of protein – membrane interactions under physiological conditions, since they allow structural and dynamic characterization at room temperature.

Our results revealed strikingly different functions encoded in the viral Spike fusion domain and thereby provide a potential calcium-dependent cell entry mechanism for SARS-CoV-2. In particular, the neutron reflectometry data demonstrated that calcium drives the protein's N-terminal to harpoon through the host membrane. However, removing calcium reorients the protein back to the lipid leaflet closest to the virus, leading to significant changes in lipid fluidity and rigidity. Molecular events leading to cell entry by SARS-CoV-2 are therefore proposed.

References

[1] Andreas Santamaria, Krishna C. Batchu, Olga Matsarskaia, Sylvain F. Prévost, Daniela Russo, Francesca Natali, Tilo Seydel, Ingo Hoffmann, Valérie Laux, Michael Haertlein, Tamim A. Darwish, Robert A. Russell, Giacomo Corucci, Giovanna Fragneto, Armando Maestro, and Nathan R. Zaccai. Strikingly Different Roles of SARS-CoV-2 Fusion Peptides Uncovered by Neutron Scattering *Journal of the American Chemical Society* (2022) 144 (7), 2968-2979

Connecting protein structure and dynamics: the neutron perspective

Frank Schreiber[a]

[a]*Institut fuer Angewandte Physik, Universitaet Tuebingen, 72076 Tuebingen, Germany*

frank.schreiber@uni-tuebingen.de

It is widely accepted that the understanding of biological function including essential transport processes requires knowledge of the structure *and* the dynamics of a system. A detailed mechanistic understanding and monitoring of protein dynamics in solution is thus highly desirable, preferably *together* with structural characterization. Here we attempt to connect the dynamics (backbone and side-chain fluctuations, interdomain motions, as well as global rotational and translational (i.e. center of mass) diffusion) to the overall structure and phase behavior in terms of clustering, crowding, crystallization, and phase separation [1]. In order to obtain a comprehensive picture, we employ a combination of small-angle scattering and quasi-elastic as well as inelastic scattering [2], along with complementary methods and simulations [3]. We also discuss the effect of interfaces on the complex phase behavior as well as the dynamics [4].

Finally, we comment on future perspectives for instrumental improvements in the context of complex systems and a simultaneous study of structure and dynamics.

Invaluable contributions by numerous collaborators are gratefully acknowledged as is financial support by the BMBF and DFG.

References

[1] M. Grimaldo et al., JPCL, 10, 1709 (2019); C. Beck et al., in preparation (2022)
F. Roosen-Runge et al., PNAS 108 (2011) 11815
[2] M. Grimaldo et al., Quarterly Reviews of Biophysics, 52, e7, 1–63 (2019)
[3] A. Girelli et al., Mol. Pharmaceutics 18 (2021) 4162, Phys. Rev. Lett. 126 (2021) 138004
[4] M. Skoda et al., J. Coll. Interf. Sci. 606 (2022) 1673, M. Fries et al., Phys. Rev. Lett. 119 (2017) 228001

Industrial Applications of Neutrons

Stora Salen

15:30-16:30

Using neutrons to unlock the properties of commercial oil additives (withdrawn or C. Boudou)

Peter J. Dowding[a], Alexander J. Armstrong[b, c], Joshua L Bradley Shaw[d] Philip J Camp[d], Beatrice Cattoz[a], Ken Lewtas[d], Thomas M. McCoy[b], Alex F. Routh[b], Rebecca J. L. Welbourne[c]

[a]Infineum UK Limited, Milton Hill, Abingdon, UK
[b]BP Institute and Department of Chemical Engineering and Biotechnology, University of Cambridge
[c]ISIS Neutron and Muon Source, Didcot, UK
[d]School of Chemistry, University of Edinburgh, David Brewster Road, Edinburgh, UK

peter.dowding@infineum.com

Additives are used in lubricant systems to minimise destructive processes in the engine (*e.g.* wear and corrosion) and to confer beneficial properties (*e.g.* reduced friction, resulting in improved fuel economy and lower environmental emissions). Around 20% of global energy is used to overcome friction with an annual global cost of ~€2.5 trillion.[1]. Organic Friction Modifiers (OFMs) are surfactants which are used as additives in engine oils to minimise friction. Over the past century an OFM mode of action has been established, where OFM adsorption at metallic surfaces is thought to form compact monolayer surface films.[2] These films are believed to form planes of low shear resistance between contacting metallic surfaces, reducing friction.

It is challenging to reproduce the conditions found within a combustion engine and probe the interfacial structure of adsorbed OFMs in-situ, and hence, much of the research has been conducted under static or mild conditions. The self-assembly of a commercial OFM, glycerol monooleate (GMO) was studied with small angle neutron scattering (SANS) as a function of surfactant concentration, solvent polarity and water content. Molecular dynamic simulation work has also indicated that the self-assembly of OFMs in the bulk solvent may influence additive adsorption and the resulting tribological properties more-so than currently thought.[3] As a result, the established OFM friction reduction mechanism remains debatable. Revealing the structure of OFMs at the solid-liquid interface in-situ will elucidate the structure-function relationships.

Neutron Reflectometry can determine the thicknesses of thin films on a sub-nm scale, and importantly can reach buried solid-liquid interfaces without suffering significant attenuation. We have used this technique to study the behaviour of GMO, adsorbed at the iron oxide-dodecane interface as a function of concentration, temperature, water content and shear, where the latter was achieved with a novel tribometer rig designed for neutron reflectometry. A discussion of our methodology and our results will be presented.

References

[1] K. Holmberg, A. Erdemir, Influence of tribology on global energy consumption, costs and Emissions, Friction 5(3): 263–284 (2017).
[2] H. Spikes, Friction modifier additives, Tribology Letters, 60(5) (2015).
[3] J. L. Bradley Shaw, P. J. Camp, P. J. Dowding, and K. Lewtas, Molecular dynamics simulations of glycerol monooleate confined between mica surfaces. Langmuir, 2016, 32, 7707–7718.

Diffraction
Industrial Applications

Abstract: 019
Oral

Dynamic cluster formation, viscosity and diffusion in monoclonal antibody solutions

Ilaria Mosca [a], *Kevin Pounot* [a,b], *Christian Beck* [a,b], *Christoph Grapentin* [c],
Olga Matsarskaia [a,b], *Tilo Seydel* [a,b] and *Frank Schreiber* [a]

[a] *Institut für Angewandte Physik, Universität Tübingen*, Auf der Morgenstelle 10, D-72076 Tübingen, Germany
[b] *Institut Max von Laue - Paul Langevin*, 71 Avenue des Martyrs, 38042 Grenoble Cedex 9, France
[c] *Lonza Group Ltd – Drug Product and Services*, Hochbergerstrasse 60G, 4057 Basel, Switzerland

mosca@ill.fr

Antibodies play an essential role in the immune response of mammals. Monoclonal antibodies (mAbs) are particularly relevant for therapeutic approaches due to their high specificity and versatility. The pharmaceutical challenge is to formulate highly concentrated antibody solutions to achieve a significant therapeutic effect, while minimizing their viscosity and keeping it under the subcutaneous injectability limit [1], thus rendering the drug administration to patients less difficult and painful. Since the understanding of macroscopic viscosity requires an in-depth knowledge on protein diffusion and dynamic cluster formation [2,3], we study the self-diffusion of five mAbs of the IgG1 subtype (produced and characterized at Lonza AG) in aqueous solution as a function of the type of antibody and of their concentration, by quasi-elastic neutron scattering (QENS). QENS allows to determine unambiguously the hydrodynamic mAb cluster size [4] and to gain information on the internal mAb dynamics. A subset of those mAbs has been also investigated using small angle neutron scattering (SANS) to obtain information on sample structure and on the nature of interactions occurring among mAb molecules. Complementary information is provided by molecular dynamics (MD) simulations and rheology measurements.

As a reference, we use polyclonal antibody (IgG from bovine serum) solutions [5], thus obtaining a comprehensive picture of mAb diffusion.

References

[1] V. Razinkov, M. J. Treuheit, and G. W. Becker. *Accelerated Formulation Development of Monoclonal Antibodies (MAbs) and MAb-Based Modalities: Review of Methods and Tools*, Journal of Biomolecular Screening 20, no. 4: 468–83 (2015).

[2] S. von Bülow, M. Siggel, M. Linke, and G. Hummer, *Dynamic cluster formation determines viscosity and diffusion in dense protein solutions*, PNAS (USA) 116, 984 (2019).

[3] M. Heinen, F. Zanini, F. Roosen-Runge, D. Fedunova, F. Zhang, M. Hennig, T. Seydel, R. Schweins, M. Sztucki, M. Antalık, F. Schreiber and G. Nägele, *Viscosity and diffusion: crowding and salt effects in protein solution*, Soft Matter 8, 1404 (2012).

[4] M. Grimaldo, F. Roosen-Runge, F. Zhang, F. Schreiber and T. Seydel, *Dynamics of proteins in solution*, Quart. Rev. Biophys. 2019, 52, e7 (2019).

[5] M. Grimaldo, F. Roosen-Runge, F. Zhang, T. Seydel, and F. Schreiber, *Diffusion and Dynamics of γ-Globulin in Crowded Aqueous Solutions*, J. Phys. Chem. B 118, 7203 (2014).

Diffraction and Magnetism

ROOM

15:30-16:30

Exotic spin states in frustrated antiferromagnet $MnSc_2S_4$

O. Zaharko[a], S. Gao[a], G. Kaur[a], H. D. Rosales[b,c], F. Gomez Albarracin[b,c], V. Tsurkan[d,e]

[a]Laboratory for Neutron Scattering and Imaging, PSI Villigen, Switzerland
[b]IFLYSIB, UNLP-CONICET, La Plata, Argentina
[c]Departamento de Fisica, Facultad de Ciencias Exactas, Universidad Nacional de La Plata, la Plata, Argentina
[d]Experimental Physics V, University of Augsburg, Augsburg, Germany
[e]Institute of Applied Physics, Chisnau, Republic of Moldova

Oksana.Zaharko@psi.ch

In antiferromagnetic spinel $MnSc_2S_4$ frustration of the nearest, J_1 and next-nearest, J_2 magnetic couplings leads to exotic spin states. We prove the existence of a spiral spin liquid [1], elucidate the multistep ordering and study the emergence of a triple-k phase in applied magnetic field [2,3] by neutron scattering. Numerical Monte Carlo simulations allow to construct an effective spin Hamiltonian accounting for these experimental findings and to identify the triple-k phase as a topologically nontrivial fractional antiferromagnetic skyrmion lattice.

References

[1] Gao S., Zaharko O., Tsurkan V., et al. Nature Physics 13, 157 (2017).
[2] Gao S., Rosales H.D., Gomez Albarracin F. A., et al. Nature 583, 37 (2020).
[3] Rosales H.D., Gomez Albarracin F. A., K. Guratinder, et al. Phys. Rev. B 105, 224402 (2022).

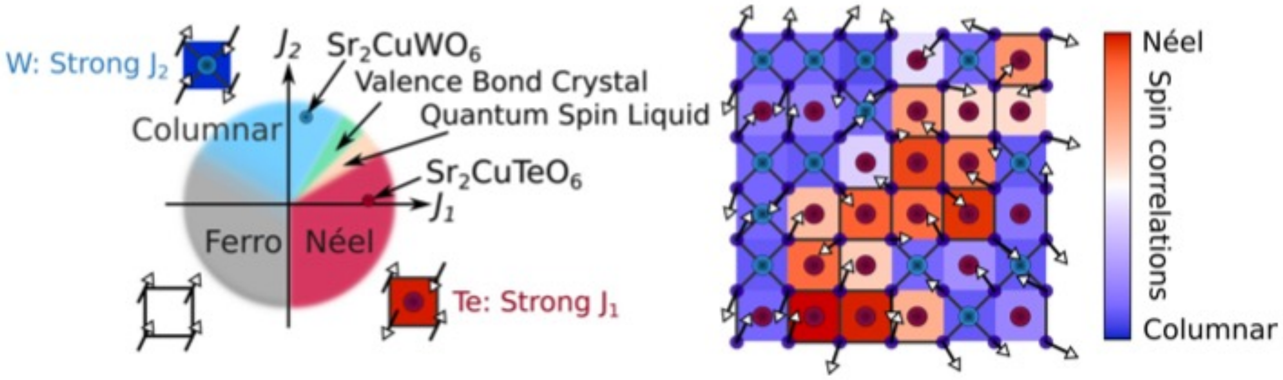
Randomness and frustration in a $S = 1/2$ square-lattice Heisenberg antiferromagnet

E. Fogh[a], *O. Mustonen*[b,c], *P. Babkevich*[a], *V. M. Katukuri*[d], *H. C. Walker*[e], *L. Mangin- Thro*[f], *M. Karppinen*[g], *S. Ward*[h], *B. Normand*[i,a] *and H. M. Rønnow*[a]

[a]*Institute of Physics, École Polytechnique Fédérale de Lausanne (EPFL), Lausanne, Switzerland,*
[b]*Department of Materials Science and Engineering, University of Sheffield, Sheffield, UK*
[c]*School of Chemistry, University of Birmingham, Birmingham, United Kingdom*
[d]*Max Planck Institute for Solid State Research, Stuttgart, Germany*
eISIS Neutron and Muon Source, Rutherford Appleton Laboratory, Didcot, UK
[f]*Institute Laue Langevin, Grenoble, France*
[g]*Department of Chemistry and Materials Science, Aalto University, Espoo, Finland*
[h]*European Spallation Source, Lund, Sweden*
[i]*Paul Scherrer Institute, Villigen, Switzerland*

ellen.fogh@epfl.ch

We explore the interplay between randomness and magnetic frustration in the series of $S = 1/2$ Heisenberg square-lattice compounds Sr2CuTe1-*x*W*x*O6. Substituting W for Te alters the magnetic interactions dramatically, from strongly nearest-neighbor (J1) to next-nearest-neighbor (J2) antiferromagnetic coupling. We perform neutron scattering measurements to probe the magnetic ground state and excitations over a range of *x*. We propose a bond-disorder model that reproduces ground states with only short-ranged spin correlations in the mixed compounds. The calculated neutron diffraction patterns and powder spectra agree well with the measured data and allow detailed predictions for future measurements. We conclude that quenched randomness plays the major role in defining the physics of Sr2CuTe1-*x*W*x*O6 with frustration being less significant.



References

[1] P. Chandra and B. Doucot, *Phys. Rev. B* **38**, 9335(R) (1988) [
2] O. Mustonen *et al.*, *Nature Comms.* **9**, 1085 (2018)
[3] V. M. Katukuri *et al.*, *Phys. Rev. Lett.* **124**, 077202 (2020)
[4] W. Hong et al., *Phys. Rev. Lett.* **126**, 037201 (2021)
[5] E. Fogh *et al.*, *Phys. Rev. B* **105**, 184410 (2022)

Chiral spin liquid ground state in YBaCo3FeO7

W.Schweika[a,b], M.Valldor[c,d], J. Reim[a,e], U.K. Rößler[c]

[a]JCNS, FZ Jülich, Germany
[b]ESS Lund, Sweden
[c]IFW Dresden, Germany
[d]Univ. Oslo, Norway
[5]Ohoku Univ., Japan

werner.schweika@ess.eu

A chiral spin liquid state is discovered in the highly frustrated, non-centro symmetric swedenborgite compound YBaCo$_3$FeO$_7$, a layered kagome system of hexagonal symmetry, by advanced polarized neutron scattering from a single domain crystalline sample. The observed diffuse magnetic neutron scattering has an antisymmetric property that relates to its specific vector chirality, which consists of three cycloidal waves perpendicular to the c-axis, forming an entity of cylindrical symmetry. Vector chirality and symmetry agree with relevant antisymmetric exchanges arising from broken spatial parity. Applying a Fourier analysis to the chiral interference pattern, with distinction between kagome sites and the connecting trigonal interlayer sites of three-fold symmetry, the chiral spin correlation function is determined. Characteristic cycloidal waves originate from the trigonal sites and extend over several periods in the kagome-planes. The chiral spin liquid is remarkably stable at low temperatures despite strong antiferromagnetic spin exchange. The observation raises a fundamental challenge, since the commonly accepted ground states in condensed matter either have crystalline long-range order or form a quantum liquid. Based on the fundamental theory of classical magnetic order, we show that a disordered ground state may arise from chirality. The antisymmetric exchange acting as a frustrating gauge background stabilizes local spin lumps. This scenario has similarities to the avoided phase transition in coupled gauge- and matter-fields for sub-nuclear particles.[1]

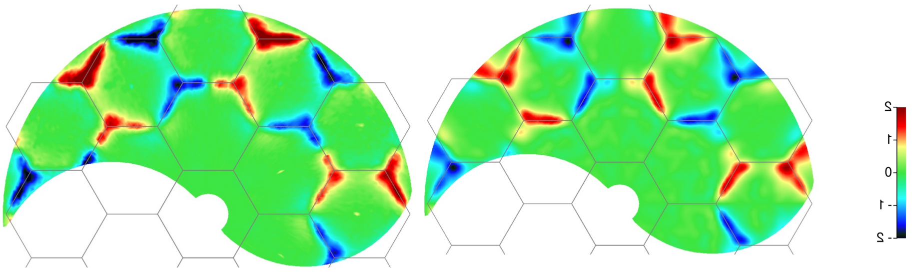


Figure 1: The anti-symmetric part of polarized diffuse neutron scattering from a single domain crystal of YBaCo$_3$FeO$_7$ at T=4K reveals a pattern that is related to vector chirality. (left) The normalized and observed chiral scattering $S_{\mathbf{Q}}^{yz}$, (right) Modeled chiral scattering based on cycloidal correlations obtained by Fourier analysis.

References

[1] W. Schweika et.al., Chiral Spin Liquid Ground State in YBaCo$_3$FeO$_7$, Phys. Rev. X **12**, 021029 (2022).

Co-existing long- and short-range magnetic order in the frustrated diamond antiferromagnet, $LiYbO_2$

Jennifer N. Graham[a,b], Navid Qureshi[a], Pascal Manuel[c], Clemens Ritter[a], Andrew R. Wildes[a] and Lucy Clark[b]

[a] Institut Laue-Langevin, Grenoble, France
[b] School of Chemistry, University of Birmingham, Birmingham, United Kingdom
[c] ISIS Neutron and Muon Source, Didcot, United Kingdom

grahamj@ill.fr

The field of frustrated magnetism explores the unconventional magnetic ground states that arise due to the competition of exchange interactions between magnetic moments [1]. Recently, a new frustrated magnet, $LiYbO_2$, has attracted attention as it contains several complex magnetic phases within an unusual stretched diamond lattice [2-4]. We have prepared a polycrystalline sample, collected high-resolution diffraction data on D2B, explored the long-range magnetic ordered states on WISH and short-range diffuse scattering on D7. Our results confirm an incommensurate helical ground state below 450 mK, with a propagation vector, $\mathbf{k} = (0.391, \pm 0.391, 0)$, and $\mu_{eff} = 1.1\mu_B$. Crucially, we find that the phase angle between the two magnetic sublattices within the stretched diamond network is 1.1π, which is in considerably better agreement with the theoretical prediction, π, than the previously reported experimental result, 0.58π [3]. We believe this difference is due to the challenges of measuring polycrystalline samples in an applied magnetic field, which required the inclusion of preferred orientation in the original analysis. Subsequently this impacted the interpretation of the intermediary magnetic phase. Therefore, between 450 mK and 1.13 K we propose a new intermediary magnetic ordered phase, where the propagation vector and moment size vary as a function of temperature. Surrounding the well-ordered magnetic Bragg peaks there is significant diffuse scattering. These magnetic diffuse features have little variation between 50 mK and 1.5 K, and analysis of spin correlations show that whilst weak, these correlations have a distinct damped oscillatory dependence. These results may indicate that a fraction of the sample remains frustrated and that the magnetic ground state is partially disordered.

References

[1] L. Clark and A. H. Abdeldaim, Ann. Rev. Mater. Res. **51**: 495-519 (2021)
[2] Y. Hashimoto *et al.* J. Phys. Condens. Matter **32** (2002)
[3] M. M. Bordelon *et al.* Phys. Rev. B. **103**, 014420 (2021)
[4] E. M. Kenney *et al.* arXiv:2205.00052 (2022)

Detection of magneto-electric multipoles

Andrea Urru,[a] Jian-Rui Soh,[b] Navid Qureshi,[c] Anne Stunault,[c] Bertrand Roessli,[d]
Henrik M. Rønnow,[b] and Nicola A. Spaldin[a]

[a]ETH Zurich, Zurich, Switzerland
[b]EPFL, Lausanne, Switzerland
[c]Institut Laue-Langevin, Grenoble, France
[d]Paul Scherrer Institut, Villigen, Switzerland

jian.soh@epfl.ch

The fundamental interaction between the neutron dipolar field and the magnetization density surrounding the scattering ion, lies at the heart of magnetic neutron diffraction. However, if the ion resides in an environment which breaks both time and spatial inversion symmetry, the current formalism for magnetic diffraction does not fully account for all the possible scattering mechanisms arising from the asymmetry of the magnetization density cloud of the scatterer [1].

In our work [2,3], we have (1) extended the theory of magnetic neutron diffraction to include these effects. Drawing analogies from the magneto-electric (ME) phenomena and standard magnetic neutron diffraction, (2) developed a framework to calculate the associated ME form factor, size of the ME multipoles and the ME propagation vector from density functional theory (DFT) calculations.

Furthermore, we have identified several material systems, which can not only host these ions but also display an ordered arrangement of these magneto-electric multipoles. Alongside our DFT calculation of the corrections to the scattering amplitudes and form factor of these multipoles, we used spherical neutron polarimetry to (3) provide evidence for the interactions between neutrons and the long-ranged order of these magneto-electric multipoles in CuO.

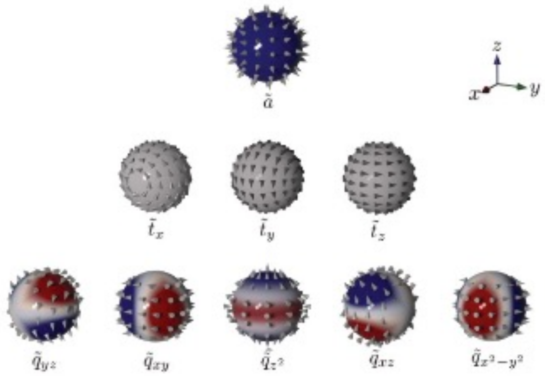


Figure 1. Full family of magneto-electric multipoles.

References

[1] S. W. Lovesey, J. Phys.: Condens. Matter 26 356001 (2014).

[2] N. A. Spaldin, M. Fechner, E. Bousquet, A. Balatsky, L. Nordström, Phys. Rev. B 88, 094429 (2013).

[3] A. Urru, J.-R. Soh, N. Qureshi, A. Stunault, H. M. Rønnow, N. A. Spaldin, in preparation.

Imaging and Engineering Materials

ROOM

15:30-16:30

Insights into martian meteorites and other planetary materials from X-ray and neutron tomography

Josefin Martell[a], Carl Alwmark[a], Robin Woracek[b] Stephen Hall[c,d], Lukas Helfen[e], Alessandro Tengattini[e], Sanna Alwmark[a,f], Ludovic Ferrière[g], Christian Bender-Koch[h], Johan Hektor[i]

[a]Department of Geology, Lund, Sweden
[b]European Spallation Source, Lund, Sweden
[c]Division of Solid Mechanics, Lund University, Sweden
[d]Lund Institute of Advanced Neutron and X-ray Science, Lund, Sweden
[e]Institut Laue Langevin, Grenoble, France
[f]Niels Bohr Institute, University of Copenhagen
[g]Natural History Museum Vienna, Austria
[h]Department of Chemistry, Copenhagen University, Denmark
[i]Department of Materials Science and Applied Mathematics, Malmö University, Malmö, Sweden.

josefin.martell@geol.lu.se

Planetary geology deals with the formation and evolution of planetary bodies in our solar system, such as the rocky planets, moons, and asteroids. Studies are often centered on meteorites and terrestrial impactites, where the latter are rocks that were formed or modified during a hypervelocity impact event. Most studies of these materials are destructive in some ways; information is gathered either by cutting thin sections for microscopy or by crushing samples for manual grain picking and elemental analyses. However, non-destructive analysis is essential when working with rare and precious samples, and is usually performed using x-ray tomography. Recently, a few studies have utilized the unique properties of neutrons to gain insight into the 3D distribution of minerals in meteorites [1] and impactites [2] and to pin-point hydrous phases in Martian meteorites [3]. There are several possible applications of combined neutron and x-ray tomography in planetary science. A few of those are described below. Detection of impactor material: Generally, meteorites have higher concentrations of nickel, chromium, and platinum group elements (e.g., iridium) compared to the Earth's crust. Iridium anomalies are of- ten used to establish extra-terrestrial impact events, the most famous being the K-Pg boundary, which marks the end of the Cretaceous period when non-avian dinosaurs became extinct [4]. Little is known about the mixing between impactor material and the host rock, and thus, resolving the 3-dimensional distribution of platinum group elements within impactites can provide insight into the impact cratering process. Sample return missions: With several upcoming sample return missions, most notably the Mars2020 sample return campaign, non-destructive analyses will be crucial both for sample characterization (e.g., mineralogy, hydrogen content) and to guide later stage destructive analyses. Hydrogen in meteorites: Hydrogen is essential to life as we know it, and consequently, locating hydrogen in extra-terrestrial materials can provide information about the aqueous history of other celestial bodies [3, 5].

In the presentation, we will elaborate on possibilities for neutron imaging in planetary science as well as discuss the results of samples already scanned at Institut Laue Langevin (ILL) and the LTH 4D imaging Lab at Lund University, in collaboration with the European Spallation Source (ESS).

References

[1] Caporali et al. (2016), Minerals 6: 14 p.
[2] Fedrigo et al. (2018), J. Imaging 4: 72 p.
[3] Martell J. et al. (2022), Sci. Adv. 8: eabn3044
[4] Alvarez L. W. et al. (1980), Science 208: 1095–1108 pp.
[5] Treiman et al. (2018), LPSC: 1993.

Characterization of triaxial deformation and hydraulic behavior of sandstones through *In Situ* testing with X-ray and neutron tomography

Fernando Vieira Lima[a], *Stephen Hall* [a,x], *Erika Tudisco*[a], *Jonas Engqvist*[a], *Robin Woracek*[b], *Alessandro Tengattini*[c], *Cyrille Couture*[c]

[a]*Lund University, Lund, Sweden*
[X]*Lund Institute of Advanced Neutron and X-ray Science, Lund, Sweden*
[b]*European Spallation Source ERIC, Lund, Sweden*
[c]*ILL, Grenoble, France*

fernando.vieira_lima@solid.lth.se

Recent advances in 3D imaging using x-rays and neutrons have enabled significant progress in understanding the formation of localized deformation and its effects on the hydraulic properties of geomaterials. Dedicated instruments now allow for simultaneous imaging using x-rays and neutrons, enabling the advantages of each modality to be exploited [1]. In this work, x-ray and neutron tomography data were acquired during coupled triaxial-permeability tests on rock to characterize the micro-scale hydromechanical behavior and relate this to the macroscopic observations obtained at the sample's boundaries.

Two coupled triaxial-permeability tests were performed on Idaho Gray sandstone samples in-situ at the NeXT instrument at the ILL. Standard, boundary measurements were made to provide the axial force, axial displacement and sample volume changes. Neutron and x-ray tomography data were acquired during the stepwise compression generating 3D image sequences with cubic voxels of 43 µm and 55 µm width, respectively. Permeability tests were performed on samples saturated with heavy water (D2O) by pressure-driven percolation of distilled water (H2O) through the sample and neutron tomography data were acquired rapidly with an acquisition time of 1 minute, generating 3D images with 170 µm cubic voxel width. Digital Volume Correlation (DVC) was performed between the images acquired at the different stages of deformation, allowing the determination of the full 3D tensor strain fields. Porosity maps were created from images for each load step. The flow analysis has been achieved using an in-house python script to determine the voxel-saturation time field, the fluid-front advance, and the flow speed field from the rapidly-acquired neutron tomography during the fluid percolation stages.

The results show that it was possible to follow the 4D flow evolution of the fluid percolation tests with neutron tomography. The H2O percolation into the saturated D2O rock was seen to follow a preferential path due to the natural heterogeneity of the sample, which could subsequently be identified in the porosity field. The flow speed measured from the images along the preferential path was higher than that estimated, considering the bulk behavior, confirming the spatial con- centration of the flow. The quantitative analysis of the sample suggests the prevalence (in frequency and intensity) of contractive strain in the first deformation step, followed by strain alleviation, in the second step, and strain localization with dilation during failure. Furthermore, a clear relationship was observed between the evolution in the flow path and the spatio-temporal evolution of the deformation in the samples, in particular the incremental volumetric strain fields.

Reference

[1] A. Tengattini et al., DOI: 10.1016/j.nima. 2020.163939.

Exploratory neutron tomography of human articular cartilage

*E Tobias B Wrammerfors [a], Elin Törnquist [a], Maria Pierantoni [a],
Alessandro Tengattini [b], Anders Kaestner [c], René in 't Zandt [d], Martin Englund [e], Hanna Isaksson [a]*

*[a]Department of Biomedical Engineering, Lund University (LU), Sweden
[b]Institut Laue-Langevin (ILL), France
[c]Paul Scherrer Institut (PSI), Switzerland,
[d]Lund Bioimaging Center, LU, Swede
[e]Clinical Epidemiology Unit, Orthopedics, LU, Sweden*

Edvin_tobias.bokvist_Wrammerfors@bme.lth.se

Neutron Tomography (NT) has shown promise in the imaging of hard skeletal tissues [1,2]. In this study, we sought to explore the potential of NT for soft skeletal tissue, with the aim of determining the medical relevance of neutron imaging and its ability to visualize detailed compositional variation in the tissue, as well as its alterations with age and disease.

We imaged human donor tissue samples (~5×5×7mm^3) of knee articular cartilage (AC) attached to subchondral bone (Fig. 1A) at the NeXT-Grenoble beamline at ILL, with the primary goal of evaluating different sample preparation techniques. This was followed up by a second experiment at the ICON beamline at SINQ, PSI to compare samples of different ages and known disease states. We further compared the outcome to laboratory X-ray microtomography (XRT, Fig. 1C), and to 9.4T magnetic resonance imaging (MRI).

Our findings show that NT images clearly visualize AC and the fatty bone marrow tissue (Fig. 1B) while XRT images only visualize the subchondral bone structure (Fig. 1C). MR T1 images (Fig. 1D) of AC are associated with the interstitial water content [3], supplementing the hydrogen content seen in NT. Comparison of preparation techniques for NT showed that D_2O soaking produced the highest intra-tissue contrast. A clear difference can be seen in the neutron attenuation gradient between healthy (young and old) and degenerated (disease) samples (Fig. 1E).

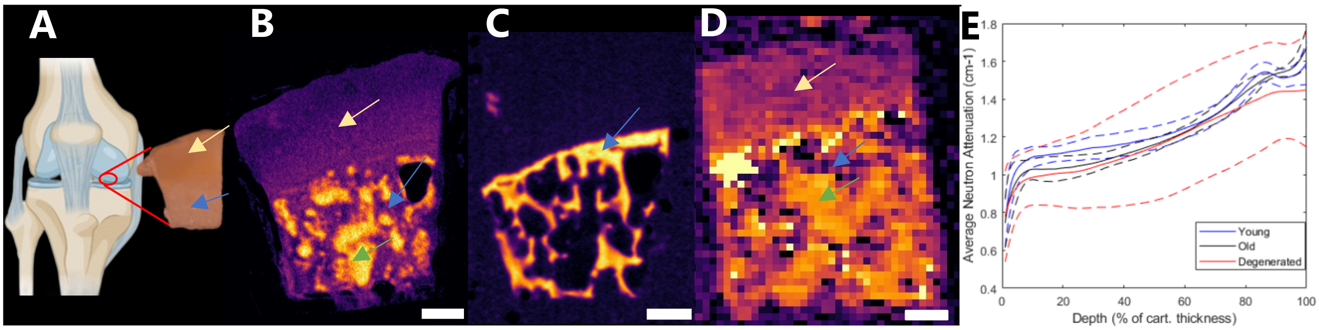


Figure 1: *(A) Location and photograph of one specimen. (B) NT slice, (C) XRT slice, and (D) MRI T1 map, containing cartilage (yellow arrows), subchondral bone (blue arrows) and marrow tissue (green arrows). Scale bar = 1mm. (E) A comparison between average hydrogen attenuation gradients of young (<50y, blue, n=5), old (>50y, black, n=6) and degenerated (red, n=3) specimens. Dashed lines show ± one standard deviation.*

References

[1] Törnquist, E., *et al.*, 2021. *Phys. Med. Biol.* **66** 135016.

[2] Guillaume, F., *et al.*, 2021. *Phys. Med. Biol.* **66** 105006.

[3] Berberat, J.E., *et al.*, 2009. *Magn. Reson. Imaging.* **27**(5):727-32.

Quantitative measurement of boron-10 using ToF transmission - How the most boring neutron experiment you could imagine became interesting

Robert Cubitt[a], *Caroline Boudou*[a]

[a]*Institut Laue-Langevin, 71 avenue des Martyrs, 38000 Grenoble, France*

cubitt@ill.fr

The quantitative estimation of boron-10 (^{10}B) content in materials is common in some industrial sectors, and in nuclear energy in particular. Various techniques exist and are often destructive (see ref 1 for a non-exhaustive review). Neutron attenuation using a white beam has been qualified as a standard test method [2] to report ^{10}B areal density. However, it relies on a delicate calibration process to account for beam hardening and sample composition. Using the D33 instrument at ILL, we have implemented a neutron time-of-flight (ToF) transmission method to measure boron-10 areal density in a simpler but yet accurate and precise way. The strength of the ToF approach lies in the acquisition of transmission data at various wavelengths in a short time.

A series of boronated aluminium bars having boron areal density ranging from 0 to 7.85mg/cm2 have been investigated. Data acquired at D33 showed a deviation from pure exponential behaviour and data fit lead to lower 10B content than expected. This phenomenon is known in the nu- clear industry as channelling [3] where there is a finite probability of the neutron missing a considerable number of absorbing *particles* (here B-10) in a non-absorbing matrix (aluminium). If we consider a variation of the areal density as a function of position, channelling is modelled with the following formula for transmission, known as the Burrus method.

We will describe the methodology, present experimental results as well as corresponding model and implications for reporting 10B areal density.

References

[1] "Boron determination – A review of analytical Methods", R. N. Sah and P. H. Brown, Micro-mechanical Journal 56, 285–304 (1997), article n°MJ971428

[2] Standard Test Method for Determination of Effective Boron-10 Areal Density in Aluminum Neutron Absorbers using Neutron Attenuation Measurements, ASTM, Volume 03.04, subcommittee E07.05, DOI: 10.1520/E2971-16R20

[3] Neutron Transmission Through Boral Impact of Channeling on Criticality, A. Machiels, 2005, technical report from the Electric Power Research Institute (EPRI): Chap 2 Burrus approach from the 1950s

Large Scale Structures and Soft Matter

ROOM

15:30-16:30

Small-angle scattering and neutron reflectometry investigation of ultra-soft colloids in two and three dimensions

Andrea Scotti [a]

[a] Institute of Physical Chemistry, RWTH Aachen University, 52056 Aachen, Germany

andrea.scotti@rwth-aachen.de

In our recent publications, we explored the phase behavior of super-soft spheres using solutions of ultra-low crosslinked poly(N-isopropylacrylamide)-based microgels as a model system [1-6].

Small-angle neutron scattering with contrast variation is used to directly access the microgel bulk modulus, K. Our measurements show that K is one order of magnitude smaller than for regular microgels synthesized using crosslinker agent [5]. In bulk, the microgel suspensions show a liquid-to-crystal transition at higher volume fraction with respect to both hard spheres and stiffer microgels. Furthermore, stable body-centered cubic (bcc) crystals are observed in addition to the expected face-centered cubic (fcc) crystals. Small-angle X-ray and neutron scattering with contrast variation allow the characterization of both the microgel-to-microgel distance, and the architecture of single microgels in crowded solutions. The measurements reveal that the stable bcc crystals depend on the interplay between the collapse and the interpenetration of the external shell of the ultra-low crosslinked microgels [1]. This peculiar phase behavior is due to strong faceting of the particles, which we determine by combining data from small-angle neutron scattering with contrast variation and computer simulations [2,3].

Then, we confine these ultra-soft microgels at air- and liquid-liquid interfaces [4]. Atomic force measurements are used to study their phase behavior. In addition, neutron reflectivity and interfacial rheology are used to probe the vertical extension of the microgels in-situ and perpendicular to the interface [6]. Once confined at interfaces, these ultra-soft spheres show a behavior in between flexible macromolecules and hard particles [4, 6].

References

[1] A. Scotti, J. E. Houston, M. Brugnoni, M. M. Schmidt, M. F. Schulte, S. Bochenek, R. Schweins, A. Feoktystov, A. Radulescu, W. Richtering. Physical Review E 102: 052602 (2020).

[2] A. Scotti. Soft Matter 17: 5548–5559 (2021).

[3] T. Höfken, C. Strauch, S. Schneider, A. Scotti. Nano Letters 22: 2412-2418 (2022).

[4] A. Scotti, S. Bochenek, M. Brugnoni, M. A. Fernandez-Rodriguez, M. F. Schulte, J. E. Houston, A. P. H. Gelissen, I. I. Potemkin, L. Isa, W. Richtering. Nature Communications 10: 1418 (2019).

[5] J. E. Houston, L. Fruhner, A. de la Cotte, J. Rojo González, A. Petrunin, U. Gasser, R. Schweins, J. Allgaier, W. Richtering, A. Fernandez-Nieves, and A. Scotti. Science Advances 8: eabn6129 (2022).

[6] S. Bochenek, F. Camerin, E. Zaccarelli, A. Maestro, M. M. Schmidt, W. Richtering and A. Scotti, Nature Communications, 13: 3744 (2022).

In-situ neutron reflectivity studies of vertical polyelectrolyte diffusion in layer-by-layer films in aqueous solutions

Annekatrin Sill[a], Heiko Ahrens[a], Olaf Soltwedel[b], Philipp Gutfreund[c], Christiane A. Helm[a]

[a]University of Greifswald, D-17489 Greifswald, Germany
[b]Technische Universität Darmstadt, D-64289 Darmstadt, Germany
[c]Institut Laue-Langevin, F-38042 Grenoble, France

annekatrin.sill@uni-greifswald.de

Layer-by-layer assembly is a widely used tool for engineering materials and coatings, [1-3] but the dynamics of the constituent polyelectrolyte chains remain poorly understood. Using neutron reflectivity, the vertical diffusion of polyanion PSS within PSS/PDADMA multilayers was probed. Up to now, we measured vertical polyelectrolyte diffusion in Layer-by-Layer films by immersing the film for a defined amount of time in the annealing solution. Then the film was dried, and a snapshot was taken. [4] With the snapshot method, one can get good results and compare them to polymer theory, but we wanted to develop an in-situ method. For the first time, the dynamics of our films were studied in-situ at the Institut Laue-Langevin (ILL) on instrument D17. We used the slab architecture with selective polyanion deuteration to study vertical polyanion diffusion. Each film consisted of a protonated and a deuterated slab. [5] Varied were the salt concentration and the temperature of the annealing solution. The diffusion constant was determined and compared to the snapshot measurements.

Chain mobility increases with the swelling of the film in water or salt solution. The in-situ measurements allowed us simultaneously to investigate the swelling behaviour of PSS/PDADMA multilayers in different solutions. Considering the increase in film thickness in D_2O, the film swells by 30%. The increase in scattering length density shows an even larger uptake of D_2O. This may indicate that existing voids in the multilayer have been filled with D_2O, or that the D_2O has a smaller molecular volume in the film than in the bulk solution. [6] Investigations using ellipsometry are performed to get complementary information.

References

[1] Zhao, Shuang, et al., *ACS nano* 13, 6 (2019): 6151
[2] Liu, Tengfei, et al., *Advanced healthcare materials* 8, 1 (2019): 1800939
[3] Mateos-Maroto, Ana, et al., *Polymers* 13, 8 (2021): 1221
[4] Sill, Annekatrin, et al., *Macromolecules* 52, 22 (2019): 9045; *Macromolecules* 54, 20 (2021): 9372
[5] Soltwedel, Olaf, et al., *Macromolecules* 43, 17 (2010): 7288
[6] Dodoo, Samuel, et al., *Physical Chemistry Chemical Physics* 13, 21 (2011): 10318; *Soft Materials* 11, 2 (2013): 157

Nanoscale wood-water interactions studied with small-angle neutron scattering

Paavo Penttilä[a], *Aleksi Zitting*[a], *Michael Altgen*[a], *Antti Paajanen*[b], *Ralf Schweins*[c], *Lauri Rautkari*[a].

[a]*Aalto University, Department of Bioproducts and Biosystems, Espoo, Finland*
[b]*VTT Technical Research Centre of Finland Ltd, Espoo, Finland*
[c]*Institut Laue-Langevin, Large-Scale Structures Group, Grenoble, France*

paavo.penttila@aalto.fi

Understanding the delicate interactions between wood cell walls and water is important for an efficient and sustainable utilization of wood in many applications. The properties of wood as a material and its processability into various products such as nanocelluloses are highly dependent on the water-accessibility of the cell wall components and their interactions with moisture. Small-angle neutron scattering (SANS) can provide new information on the nanoscale structure of wood cell walls and the role of water as a structural component in it. Analysis of SANS data obtained from wood samples is possible using the WoodSAS model [1], which provides information on the lateral size and packing distance of cellulose microfibrils (diameter 2-3 nm). With the help of contrast variation provided by polyethylene glycol, one of the scattering contributions was linked to the diameter of cellulose microfibril bundles (10 to 20 nm) [2].

Most recently, we have utilized time-resolved SANS to study the location and transfer of water in the hierarchical structure of wood cell walls. We could observe how liquid water is exchanged within the bundles of cellulose microfibrils, with most of the exchange taking place in the scale of minutes and rather homogeneously [3]. In another work, we followed the drying of a fully wet wood sample under ambient conditions and could link the deswelling of the microfibril bundles with a decreasing diffusivity of water [4]. The results demonstrate the possibilities of modern SANS instruments in time-resolved studies, and highlight their value in providing fundamental new information on the nanoscale structure and water-interactions of wood.

References

[1] Penttilä, P.A., Rautkari, L., Österberg, M., & Schweins, R. Small-angle scattering model for efficient characterization of wood nanostructure and moisture behaviour. Journal of Applied Crystallography 52, 369-377 (2019). DOI: 10.1107/S1600576719002012

[2] Penttilä, P.A., Altgen, M., Awais, M., Österberg, M., Rautkari, L., & Schweins, R. Bundling of cellulose microfibrils in native and polyethylene glycol-containing wood cell walls revealed by small-angle neutron scattering. Scientific Reports 10, 20844 (2020). DOI: 10.1038/s41598-020-77755-y

[3] Penttilä, P.A., Zitting, A., Lourençon, T., Altgen, M., Schweins, R., & Rautkari, L. Water-accessibility of interfibrillar spaces in spruce wood cell walls. Cellulose 28, 11231-11245 (2021). DOI: 10.1007/s10570-021-04253-3

[4] Zitting, A., Paajanen, A., Rautkari, L., & Penttilä, P.A. Deswelling of microfibril bundles in drying wood studied by small-angle neutron scattering and molecular dynamics. Cellulose 28, 10765-10776 (2021). DOI: 10.1007/s10570-021-04204-y

Localization of dye molecules in surfactant assemblies via SANS contrast variation

Wenke Mueller[a]*, Ralf Schweins*[a]*, Bernd Noecker*[b]*, Klaus Huber*[c]
[a]*Institut Laue-Langevin, Grenoble, France,*
[b]*KAO Germany GmbH, Grenoble, Germany,*
[c]*University of Paderborn, Paderborn, Germany*

muellerw@ill.fr

Organic azo dyes are interesting building blocks in supramolecular chemistry. The responsivity of the dyestuff spectrum to changes in their chemical environment is often used to monitor alterations in a solution state by means of simple UV/vis-spectroscopy[1]. However, little attention was paid to the morphology of dye/surfactant assemblies[2]. Small-angle neutron scattering (SANS) with the technique of contrast variation permits the elucidation of assembly-morphology and the unambiguous localization of dye-molecules within surfactant micelles. We studied the interaction between the commercial, anionic azo dye Blue and the cationic surfactant DTAB in an alkaline buffer solution. Solutions of Blue and DTAB phase-separate above a Blue:DTAB ratio of 1:2.5. Below this ratio, stable solutions form and the absorption spectrum of Blue depends on the concentration of DTAB (**Fig. 1, left**). To better understand underlying mechanisms, the morphology of assemblies in the 1-phase region was studied with SANS. Moving from the precipitation threshold to the surfactant-rich side leads to a shrinking of assembly size and a reduction in its anisotropy. This is indicated in **Fig. 1, right**. SANS contrast variation was performed by matching DTAB to the solvent and observing the scattering signal arising from Blue only. For all sample compositions, we unambiguously located Blue on a surface-layer of the dye-surfactant micelle. This work demonstrates the feasibility of SANS contrast variation for dye/surfactant-systems and relates findings to UV/vis spectroscopic investigations.

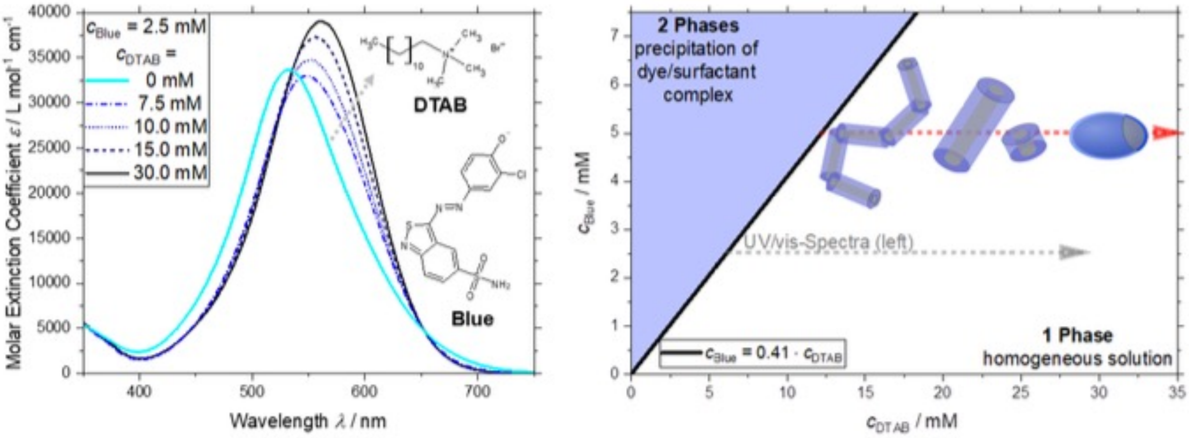


Figure 1: Left: Change of Blue UV/vis spectrum upon addition of DTAB. Right: Phase diagram.

References

[1] A. R. Tehrani-Bagha, K. Holmberg, Materials 2013, 6, 580–608.
[2] A. Kutz, G. Mariani, F. Gröhn, Colloid Polym Sci 2016, 294, 591–606.

Nanostructure in amphiphile-based deep eutectic solvents

Iva Manasi[a], Ralf Schweins[b] and Karen J. Edler[a,c]

[a]Department of Chemistry, University of Bath, Claverton Down, Bath BA2 7AX, United Kingdom
[b]Institut Laue-Langevin, CS 20156, 38042 Grenoble Cedex 9, France
[c]Centre for Analysis and Synthesis (CAS) Lund University, Lund, 221 00 Sweden

im554@bath.ac.uk

Deep eutectic solvents (DES) are mixtures of hydrogen bond donors and acceptors that form strongly hydrogen-bonded room temperature liquids. DES have tunable physicochemical properties making them viable green solvents. Recently, a deep eutectic system based on amphiphilic surfactant N-alkyl-N,N-dimethyl-3-ammonio-1-propanesulfonate (C12 & C14; sulfobetaine) and (1S)-(+)-10-camphor-sulfonic acid in the molar ratio 1:1.5 has been reported [1]. These are highly viscous solvents, viscosity ~3 Pa s, with melting point below 0°C. Nanostructuring can be expected in these solvents due to the nature of their components, similar to that observed in long alkyl chain ionic liquids [2], which can also be the cause of their extremely high viscosity.

In this work we have investigated the native nanostructure in the DES comprising C12-C18 alkyl chain sulfobetaines and how it interacts with polar and non-polar species, water and dodecane respectively, using small angle neutron scattering. By using contrast variation to highlight the relative position of the solvent components and additives, we can resolve the structure of the solvent and how it changes upon interaction with water and dodecane. Scattering from the neat DES shows structures corresponding to the self-assembly of sulfobetaines; the size of the structure increases as the alkyl chain length of the sulfobetaines increases. Water and dodecane interact with the hydrophilic and hydrophobic moieties in the DES structure, primarily the sulfobetaine, thereby swelling and solvating the entire structure. The extent of the shift of the peak position, and the swelling, depends on concentration of the additive. The solution phase organisation and the interaction of polar and non-polar species as observed here, has the potential to affect the ordering of inorganic or polymeric materials grown in such solvents, paving new avenues for templating applications.

References

[1] Cardellini et al *RSC Adv* **2015**, 5, 31772-31786.

[2] Cabry et al *Faraday Discussions* **2018**, *206*, 265-289

Spectroscopy and Functional Materials

ROOM

15:30-16:30

Anionic exchange membranes for fuel cells: insight into ion, polymer, and water dynamics from neutron spectroscopy

F. Foglia[a], Q. Berrod[b], A. J. Clancy[a], K. Smith[c], G. Gebel[b], V. García Sakai[d], M. Appel[e], J.-M. Zanotti[f], M. Tyagi[g,h], N. Mahmoudi[d], T. S. Miller[c], J. R. Varcoe[h], A. P. Periasamy[i], D. J. L. Brett[c], P. R. Shearing[c], S. Lyonnard[b], P. F. McMillan

[a]Department of Chemistry, Christopher Ingold Laboratory, UCL, London, UK
[b]Université Grenoble Alpes, CNRS, CEA, IRIG-SyMMES, Grenoble, France
[c]Electrochemical Innovation Lab, Department of Chemical Engineering, UCL, London, UK
[d]ISIS Neutron and Muon Source, Rutherford Appleton Laboratory, Chilton, UK
[e]Institut Laue Langevin, Grenoble, France
[f]Laboratoire Léon Brillouin, Université Paris-Saclay, CEA Saclay, Gif-sur-Yvette Cedex, France
[g]NIST Center for Neutron Research, Gaithersburg, MD, USA
[h]Department of Materials Science and Engineering, University of Maryland, College Park, MD, USA
[i]Department of Chemistry, University of Surrey, Guildford, UK

f.foglia@ucl.ac.uk

Ion conducting polymer membranes are designed for applications ranging from separation and dialysis, to energy conversion and storage technologies. A key application is in fuel cells, where the semi-permeable polymer membrane plays several roles. In a fuel cell, the polymer membrane permits the selective transport of H^+ or OH^- to enable completion of the electrode half-reactions, plays a major role in the management of water that is necessary for the conduction process and is a product in the reactions, and provides a physical barrier against leakage across the cell. All of these functions must be optimised to enable high conduction efficiency under operational conditions, including high temperatures and aggressive chemical environments, while ensuring a long lifetime of the fuel cell. Polymer electrolyte membranes used in current devices only partially meet these stringent requirements, with ongoing research to assess and develop improved membranes for a more efficient operation and to help realise the transition to a hydrogen-fuelled energy economy. A key fundamental issue to achieving these goals is the need to understand and control the nature of the strongly coupled dynamical processes involving the polymer, water and ions, and their relationship to the conductivity, as a function of temperature and other environmental conditions. This can be achieved by using neutron scattering techniques that give access to information across a wide range of timescales. Here QENS is applied over a wide range of timescales to disentangle the water, polymer relaxation and OH^- diffusional dynamics in commercially available anion exchange membranes designed for selective anion transport across different technology platforms, using the concept of serial decoupling of relaxation and diffusional processes to analyse the data. The insights provided by these data [1] offer a guide for the design of new devices, where tuning the membrane nanostructure would result in activation of the anionic hopping mechanism, providing improved performance over a wide range of operational conditions.

References

[1] F. Foglia et al., Nature Materials (2022) 21 (5), 555.

A combined experimental and computational study of oxide ion conductors

Bettina Schwaighofer [a,b], *Miguel Angel Gonzalez* [a], *Ivana Radosavljević Evans* [b]

[a]*Institut Laue Langevin, Grenoble, France*
[b]*Durham University, Durham, UK*

schwaighofer@ill.fr

Solid oxide ion conductors are remarkable materials with significant market potential due to their importance in technological applications like oxygen sensors, fuel cells and separation membranes. A crucial factor for advancement of their applicability is the development of new oxide ion conductors with higher conductivities at lower temperatures. Detailed knowledge of the structural characteristics, resulting conduction pathways and dynamics in these materials is a key requirement for improving their properties. Quasi-elastic neutron scattering is an excellent method for studying solid state diffusion and allows the observation of oxygen dynamics on a microscopic timescale. Combined with ab-initio molecular dynamics simulations, it can provide a comprehensive insight into diffusion processes on the atomic scale. We used this combined approach to investigate and compare the different oxide ion dynamics in two isostructural materials: Bi0.852V0.148O1.648 and Bi0.852P0.148O1.648, and account for the superior performance of the vanadate. Using the backscattering spectrometer IN16b and the time-of-flight spectrometer IN5 at the ILL allowed direct observation of dynamics on the nanosecond and picosecond timescales, and analysis in conjunction with molecular dynamics simulations allowed us to elucidate the nature of the observed dynamics as well as the structural characteristics important for oxide ion conduction in these doped bismuth oxides.

Endofullerenes: dynamics in confinement - $CH_4@C_{60}$ Study

Mohamed Aouane[a], George Bacanu[b], Mark Walkey[b], Elizabeth Marsden[b], Sally Bloodworth[b],
Richard Whitby[b], Malcolm Levitt[b] and Stéphane Rols[a]

[a]Institut Laue-Langevin, Grenoble, France
[b]University of Southampton, United Kingdom

aouanem@ill.fr

Endofullerenes are substances in which small molecules or atoms are encapsulated in highly symmetrical cages of carbon atoms. Each encapsulated atom or molecule behaves as a textbook example of a quantized particle-in-a-box. The newest and largest member of the endofullerene family tree, namely $CH_4@C_{60}$, is the first organic molecule trapped inside the cage and the main interest of this abstract. The use of various spectrometers at ILL, namely PANTHER, IN5 and IN1-LAGRANGE, have allowed us to probe a large energy window up from 1 to 100 meV. The goal from these experiments was to probe the rotational and translational states of the confined CH_4 molecule, and observe the coupling between said states due to the confinement. A novel result that we have observed in this endofullerene, was the effect of the I_h symmetry of the C_{60} on the rotational states of the CH_4 using inelastic neutron scattering, an effect that was absent in previous endofullerene studies. We have also performed QENS measurements, using PANTHER, that have allowed us to observe slight differences in the rotational dynamics of both the C_{60} and CH_4, confirming the stronger guest-host coupling that we observed in our INS measurements.

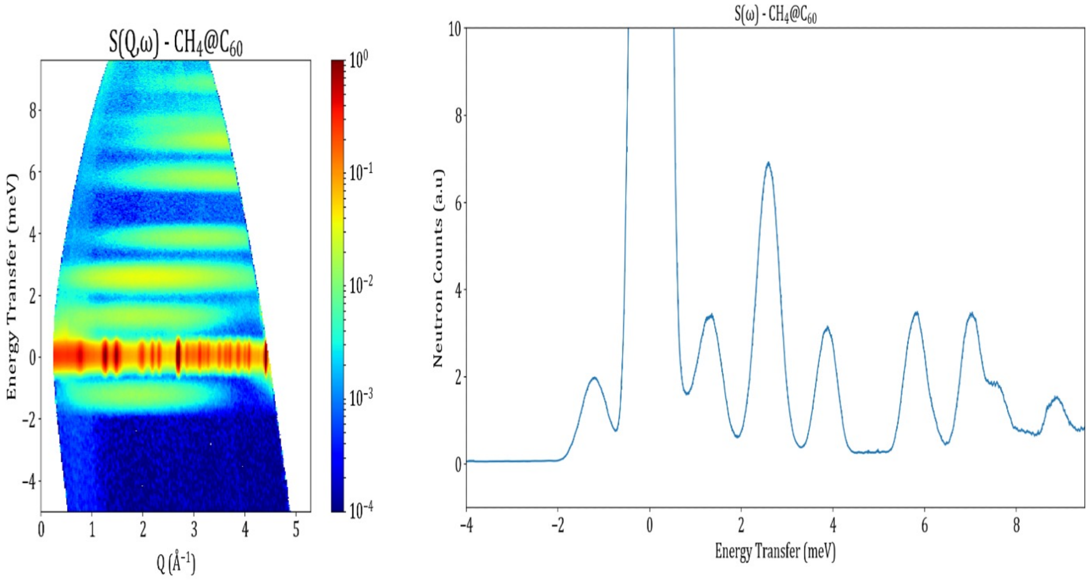


Figure 1: Inelastic neutron scattering results from the $CH_4@C_{60}$ measurements

Hydrogen mobility and reactivity in MoS_2 catalyst

Peter Fouquet[a], Vitalii Kuznetsov[ab], Leran Lu[a], Michael M. Koza[a], Wiebke Lohstroh[c], Detlef Rogalla[d], Hans-Werner Becker[d], Thomas Strunkus[e], Alexei Nefedov[f], Eva Kovacevic[g], Franziska Traeger[b]

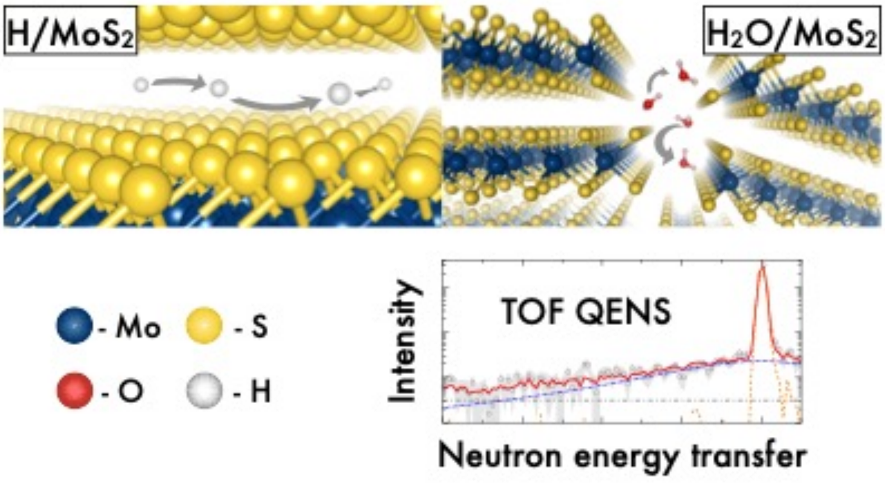
[a]Institut Laue-Langevin, Grenoble, France,
[b]Westfälische Hochschule, Recklinghausen, Germany,
[c]TUM, FRM II, Garching, Germany,
[d]RUBION, Ruhr-Universität Bochum, Bochum, Germany,
[e]Universität Kiel, Germany,
[f]KIT, Eggenstein-Leopoldshafen, Germany,
[g]GREMI, Orléans, France

fouquet@ill.fr

Catalyst materials are an essential component in hydrogen production and for the operation of fuel cells. Today, however, they are mostly based on platinum, which is expensive and rare. Molybdenum sulfide, MoS_2, which is more abundant and significantly cheaper, has shown interesting catalytic activity and has been a focus point of research in recent years [2]. MoS_2 is a van-der-Waals bonded 2d material that, theoretically, provides a high density of active sites, but little is known about the mobility and the reaction steps of hydrogen and water in MoS_2.

In this presentation we will discuss our recent studies on hydrogen mobility and reactivity in MoS_2 [1]. To shed light on the possible pathways for improving the performance of MoS_2 and other 2d catalyst materials, we have studied the diffusion of hydrogen and water by means of neutron scattering and X-ray photoelectron spectroscopy combined with nuclear reaction analysis and molecular dynamics simulations.

We observed a very fast hydrogen diffusion parallel to the basal planes and a very slow diffusion perpendicular to the MoS_2 basal planes. Water, on the other hand, cannot penetrate the perfect crystal, but can intercalate within volume defects, where it can access edge sites of the material. MD simulations were essential for the identification of the contributions of the different hydrogen species ($H^{(+)}$, H_2, H_2O) to the chemistry in MoS_2.



References

[1] D. Merki et al, Chemical Science **2**, 1262, (2011), Y. Cao, ACS Nano **15**, 11014, (2021), J. Aliaga et al., Molecules **24**, 4631, (2019), J. Xu et al., Chemistry – A European Journal **27**, 15992, (2021), Q. Yang, et al., J. Phys. Chem. C **123**, 10917, (2019), G. Li, et al. ACS Nano **14**, 1707–1714, (2020).
[2] V. Kuznetsov et al. PCCP **23**, 7961-7973, (2021).

Vibrational properties of $A\mathrm{PbI}_3$: from harmonic vibrations to a phonon liquid

Rasmus Lavén[a,], Michael M. Koza[b], Garrett E. Granroth[c], Fanni Juranyi[d], Lorenzo Malavasi[e],*
Aymen Yangui[a], Maths Karlsson[a]

[a]Department of Chemistry and Chemical Engineering, Chalmers University of Technology, Sweden
[b]Institut Laue-Langevin, France
[c]Neutron Scattering Division, Oak Ridge National Laboratory, USA
[d]Paul-Scherrer Institute, Switzerland
[e]Department of Chemistry, University of Pavia, Italy

rasmus.laven@chalmers.se

Metal halide perovskites (MHPs) are currently attracting a large interest due to potential applications in, for example, photovoltaics and light-emitting devices. Importantly, these materials are characterized by coherent electronic band transport expected from crystalline semiconductors, but phonon dynamics typical of liquids. This has been argued to be responsible for large polaron formation and screening of charge carriers, leading to defect tolerance, moderate charge carrier mobility, and radiative recombination properties [1]. While this observed “phonon melting” happens at the onset temperature for the rotational dynamics of the organic cation ($CH_3NH_3^+$) in the prototypical MHP $CH_3NH_3PbI_3$, a previous Raman study on the all-inorganic MHP $CsPbBr_3$ suggests that it is a generic feature of the soft lead halide framework [2]. Accordingly, the nature and effect of the organic cation and perovskite lattice dynamics on the structural and optical properties of MHPs remain not fully understood.

Motivated by the lack of understanding pertaining to the role of organic cation and perovskite lattice dynamics on the optical properties of MHPs, we here report a detailed inelastic neutron scattering study focusing on the vibrational properties of the all-inorganic MHP $CsPbI_3$. The orthorhombic δ-phase of $CsPbI_3$ is found to feature a harmonic and almost temperature-independent lattice dynamics, that is in well agreement with harmonic lattice dynamics calculations, from 10 K all the way up to the orthorhombic-to-cubic phase transition at around 600 K. In contrast, the cubic perovskite phase of $CsPbI_3$ is found to feature a highly anharmonic and overdamped vibrational dynamics, with no features in the vibrational density of states (vDOS). Upon passing through the orthorhombic-to-cubic phase transition at around 600 K, there is a sharp increase in low-energy scattering which is interpreted as an increase in overdamped or relaxational dynamics, that is mainly attributed to displacements of the Cs^+ cations. Analyzing the momentum transfer dependence of the inelastic scattering reveals atomic correlations, meaning that the highly overdamped optical phonons still retain their spatial correlations in the perovskite phase, even though they are heavily overdamped in frequency space. Lastly, we compare the vibrational dynamics of the all-inorganic $CsPbI_3$ to its hybrid counterparts $(CH_3NH_3)PbI_3$ and $(CH[NH_2]_2)PbI_3$. The hybrid materials are found to exhibit an even larger overdamping of the optical phonons, and a linear low energy vDOS that is typical of liquids. This is explained due to the organic cation rotations which creates further dynamics disorder and leads to an even shorter lifetime of phonons.

References

[1] H. Zhu *et al.*, Science **353**, 1409–1413 (2016).

Imaging for Archeology and Material Science

Stora Salen

09:00-10:30

Using neutrons to explore processes in geomaterials

Stephen Hall[a,b]

[a]*Division of Solid Mechanics, Lund University, Lund, Sweden*
[b]*LINXS Institute of advanced Neutron and X-ray Science, Lund, Sweden*

stephen.hall@solid.lth.se

Neutrons provide the ideal probe to address many research questions concerning geomaterials, i.e. soils, rocks, concrete and related materials. Firstly, in many applications involving geomaterials, such as pollutant transport, water resource engineering, hydrocarbon production and CO2 sequestration, the presence, location and transport of water and other hydrogen-rich fluids is key. Given the strong interaction of neutrons with hydrogen and the lower interaction with the mineral constituents of the geomaterials, neutron imaging can provide clear details on fluid distributions. Furthermore, the low neutron interaction with many metals, e.g., aluminium, also facilitates imaging of samples under pressure/temperature conditions mimicking the operational environments in foundations or oil/gas reservoirs. Consequently, recent advances in neutron imaging coupled with adapted experimental devices are providing great opportunities for geoscientists in this context, enabling, for example, high resolution, 3D timelapse imaging of fluid infiltration/migration processes.

Neutrons also provide the possibility to probe deformation processes in geomaterials, through imaging and diffraction. Digital Volume Correlation can be used, for example, to analyse deformation in 3D based on tomographic images acquired during in-situ deformation experiments. Alternatively, neutron diffraction scanning can be used to measure strain and, thus, stress fields in, for example, sand due to the crystalline nature of the constituent grains. This opens the possibility to measuring both stress and strain fields simultaneously if coupled with, for example, Digital Image Correlation.

Examples will be presented of recent research on hydraulic, mechanical and hydromechanical processes using neutron imaging and diffraction, including the effects of heterogeneity and mechanical deformation on fluid flow in rocks and coupled stress and strain fields in sand during deviatoric loading, as well as freezing processes in concrete and other examples.

Dual modality neutron and X-ray tomography data from skeletal tissues

E. Törnquist [a], S. Le Cann [a,b], D. R. Raina [c], A. Tengattini [d,e], E. Andò [e], N. Lenoir [d,e], J. Hektor [f],
E. Tudisco [g], M. Tägil [c], S. H. Hall [h], H. Isaksson [a]

[a] Biomed. Eng., Lund University (LU), Sweden,[b] CNRS, UPEC Créteil, France
[c] Orthopedics, LU, Sweden ,[d] Institut Laue-Langevin, Grenoble, France
[e] Univ. Grenoble Alpes, CNRS, Grenoble INP, 3SR, France
[f] LUNARC - Centre for Scientific and Technical Computing LU, Sweden
[g] Geomech. Eng., LU, Sweden, [h] Solid Mechanics, LU, Sweden

hanna.isaksson@bme.lth.se

Background

Bone is a complex hierarchically structured tissue whose mechanical properties are optimised for fracture resistance in healthy bone. Degenerative disorders and trauma reduce bone strength and fracture resistance and often result in the need for medical interventions involving implants. Neutron tomography (NT) has shown promising results as an alternative technique to X-rays tomography (XRT), which is the state-of-the-art in bone research, for evaluation of bone-implant integration in close proximity to a metallic implant due to the lack of image artifacts in the neutron data (1,2).

Aims/Objectives

The current study aimed at elucidating the complementary nature of neutron and X-ray tomography for evaluating the bone ingrowth around a metal implant.

Methods

Specimens consisted of distal rat tibiae with hollow titanium screws implanted and left to integrate for 6 weeks. The specimens were dried and imaged with high resolution NT and XRT at the D50 NeXT beamline at Institute Laue-Langevin, France (3). Using image registration as described in (4,5), where the dual histogram is used to identify phases and improve registration, the complementary nature of the modalities was investigated by looking at differences in featured structures and image quality in terms of visualisation accuracy.

Findings and Conclusions

Analysis of the dual histogram and phase segmentation obtained from the registration showcases the complementary nature of the two modalities in that different structures are visualised differently in NT and XRT. Due to the difference in interaction between neutrons and matter, soft tissue (which is high in hydrogen) is better captured with NT than with XRT. Sever artefacts around the metallic implant reduced the contrast between structures and background in XRT. Higher spatial resolution in XRT yields more accurate visualisation of small structures away from the implant. However, lack of artefacts close to the implant in NT enables clearer visualisation of the bone-implant ingrowth and immediate interface.

Figure 1. Representative sagittal (i) and transverse (ii) slices of registered NT image (a), XRT image (b), and checkerboard comparison of both (c). Both NT and XRT are displayed in the grey scale interval 0-255.

References

1. Isaksson H, et al. Bone (2017); 2. Le Cann S, et al. J Mech Behav Biomed Mater (2017); 3. Tengattini A, et al. Nucl Inst Methods Phys Res A (2020); 4.Tudisco E, et al. Meas Sci Technol. (2017); 5.Roubin E, et al. Cem Concr Compos (2019)

Laue and time-of-flight neutron diffractive imaging methods for 3D grain mapping of polycrystalline materials

C. B. Larsen[a], S. Samothrakitis[a], N.F. Gade-Nielsen[b], S. Schmidt[c]

[a]Paul Scherrer Institute, Villigen, Switzerland
[b]Technical University of Denmark, Kgs. Lyngby, Denmark
[c]European Spallation Source ERIC, Lund, Sweden

camilla.larsen@psi.ch / stavros.samothrakitis@psi.ch

For polycrystalline materials, key material properties including strength, deformation behavior, magnetic susceptibility, weldability and stress corrosion cracking resistance depend significantly on the texture of the crystalline microstructure. Conventional assessment of texture is either limited to thin surface regions or it is destructive while only probing small fractions of a bulk specimen. Only high energy X-ray diffraction at synchrotron sources and neutrons enable quantitative studies of bulk texture. Here, we report how transformative progress in advanced Laue three-dimensional neutron diffraction tomography and six-dimensional X-ray and neutron diffraction enable to map several hundred grains and, thus, allows grain orientation assessment in the volume of centimeter-sized samples with statistical significance. Laue 3DNDT is performed with short exposure times and efficient experimental processes, utilizing a white thermal neutron beam, while the 6DXND method makes use of a wavelength-resolved beam, in a time-of-flight mode, to provide sub-grain levels of information. The non-destructive nature of both methods will support in-situ studies, while future improvements in spatial resolution shall provide with more accurate grain morphology in corresponding microstructure studies.

Neutrons for Europe
USER MEETING
2022

Diffraction for Energy and Sustainability

ROOM

09:00-10:30

Quantification of operation-driven active material losses in Li-ion batteries using neutron diffraction

Anatoliy Senyshyn[a]

[a]Heinz-Maier Leibnitz Zentrum, Technische Universität München, German

anatoliy.senyshyn@frm2.tum.de

Right since their invention, the energy storage media based on Li-ion technology have significantly gained in popularity for a wide field of applications ranging from supplying portable devices to electrification of drivetrains and local storage of renewable energy. Such a rapid "invasion" is caused by the unique features of Li-ion technology such as high energy and power densities (both gravimetric and volumetric), low self-discharge when not in use, tiny memory effect etc. Despite the overall success of Li-ion technology further progress permanently demands for lower-cost, longer-life, higher energy/power density batteries resulting in active development and research in this field. Nowadays modern Li-ion batteries are sophisticated electrochemical devices, possessing numerous degrees of freedom along with complicated geometries of the electrode integration. This along with the need to minimize the risks for possible materials oxidation, electrolyte evaporation, cell charge changes etc. calls for the application of experimental techniques capable to reveal "live" information about processes occurring inside the cell. In such instance neutron scattering is already a well-established experimental toolbox for the characterization of Li-ion batteries. *In situ* and *in operando* experiments with neutrons are carried out on various cells with different types, designs, chemistries and shapes (either lab-based or commercial) depending on the research needs.

Operation of typical Li-ion battery involves big number of different processes occurring on variety of scales and times, whose interplay is not entirely systematized and variety of cell chemistries and designs further adds to the complexity. Such a complexity of modern Li-ion batteries often result in spatial inhomogeneity of current, lithium or electrolyte distribution, which are difficult to model and to quantify non-destructively. However, non-uniformities of cell parameters are affecting cell performance, cycling stability, safety and cell aging\fatigue further leads to the stabilization of heterogeneous state in Li-ion batteries [1]. In the current contribution, the aspect of active material losses occurring during cell aging and its quantification using neutron diffraction will be addressed [2]. Examples of studies illustrating the quantification of fatigue driven losses of lithium inventory, electrode materials and liquid electrolyte will be presented along with the applications of spatially-resolved neutron diffractions towards localization of the fatigue-driven heterogeneities in cylinder-type Li-ion batteries [3].

References

[1] D. Petz et al., J. Power Sources, 448 (2020) 227466.
[2] M.S.D. Darma et al., ACS Appl. Energy Mater., 4 (2021) 9982-10002.
[3] M. Mühlbauer et al., J. Power Sources, 475 (2020) 228690.

Neutrons to understand improvement in battery performance on electrode modification

Neelima Paul[a]

[a] *Heinz Maier-Leibnitz Zentrum, Technische Universität München, D-85748 Garching, Germany*

neelima.paul@frm2.tum.de

Electric vehicles are on the way to replace gasoline-powered vehicles in our near future. To power such vehicles, Li-ion batteries with very high storage capacities are necessary, so that these cars travel sufficiently large distances before needing a recharge at charging stations. Moreover, these batteries should have long lifetimes so that they can be charged and discharged several times before needing a replacement. Now energy storage capabilities, rate performances and cycling stabilities of Li-ion batteries are strongly dependent on the electrode materials. As practical energy densities accessible with cathode materials are already close to their theoretical limits with the currently available electrolytes, improvements or replacement of anode materials becomes more paramount than the cathode materials. The widely used graphite anode is stable, but offers low energy density, and suffers from side reactions, which are severe at higher charging rates and low temperatures, leading to eventual capacity fading. Titanium oxide anodes offer much longer life cycles, but have even lower energy densities. Li metal anodes offer the highest possible energy density, but are prone to dendrite formation and thus not preferred for safety reasons. Si anodes offer second highest possible energy densities, but suffer from large volume changes leading to fast capacity fading. With help of several analytical methods, including neutron based methods; this contribution will show how battery performances can be enhanced by either altering the anode morphology, or preparing composite anode mixtures, or by applying coatings to anode surfaces.

The first neutron investigation of multiple phase transitions in Prussian White

Ida Nielsen[a], D. Dzodan[a], D. O. Ojwang[b], H. L. B. Boström[c], and W. R. Brant[a]

[a]*Department of Chemistry – Ångström Laboratory, Uppsala University, Uppsala Sweden.*
[b]*TioTech AS, Nesttun, Norway.*
[c]*Max Planck Institute for Solid State Research, Stuttgart, Germany.*

ida.nielsen@kemi.uu.se

A promising cathode material for Na-ion batteries is the Fe-based vacancy-free Na-rich Prussian Blue Analogue (PBA) known as Prussian White (PW) Na2Fe[Fe(CN)6]·zH2O. Na-based PBAs adopt different structures depending on the Na+ and H2O content (Figure 1). For example, dehydration of PW results in a phase transition from *P21/n* to *R-3* symmetry and an 18% volume contraction. Desodiation of the dehydrated PW subsequently results in a transformation to *Fm-3m* symmetry and a volume expansion (~17%). This large change in volume is not observed during desodiation of the hydrated structure. It is clear that H2O plays a vital role for structural stability1 and yet its presence is highly damaging in non aqueous systems. Despite the large volume of work done on PBAs, the use of X-ray diffraction to study the structure2,3 has resulted in a limited understanding of the mechanism behind the observed phase transitions.4 Here, we show symmetry-mode refinements using ISODISTORT5 against neutron diffraction data of several PW samples before and after dehydration and the subsequent re-hydration. For the first time, a phase transition is observed at 30 °C from *P21/n* to *R-3* due to a change in tilting from *a-a-b+* to *a- a-a-* without the corresponding change in volume or H2O loss. These findings imply that the tilt system is not necessarily dependent on the composition. Dehydration results in a severe tilting of the Fe octahedra along with displacements of the Na positions while retaining the *R-3* symmetry and *a-a-a-* tilt pattern. Upon re-hydration, the degree of tilting of the Fe octahedra reverts back to the pristine structure with H2O and Na at similar positions. However, a severe broadening of the re- flections implies a loss of crystallinity during this process. Understanding the position and orientation of H2O during these processes will help to understand the structural changes observed as well as material properties that depend on H2O such as the voltage output.

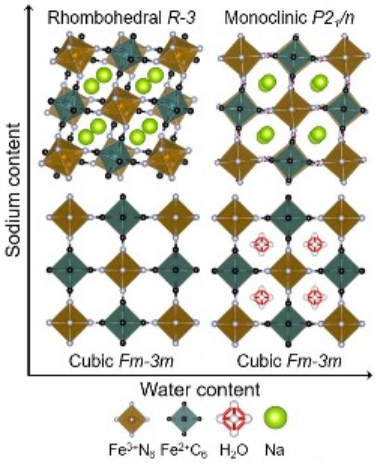


Figure 1: Phase diagram for low valency iron-based PW ($Na_2Fe[Fe(CN)_6]$)

References

[1] Guo, X. *et al. Chem. Mater.* 31, 5933–5942 (2019).
[2] Ivanov, V. D. *Ionics (Kiel).* 26, 531–547 (2020).
[3] Boström, H. L. B. *et al. Chem. Commun.* 55, 10230–10233 (2019).
[4] Boström, H. L. B. *et al.* J. Mater. Chem. C. Advance Article (2022). 5. Campbell, B. J. *et al. J. Appl. Crystallogr.* 39, 607–614 (2006).

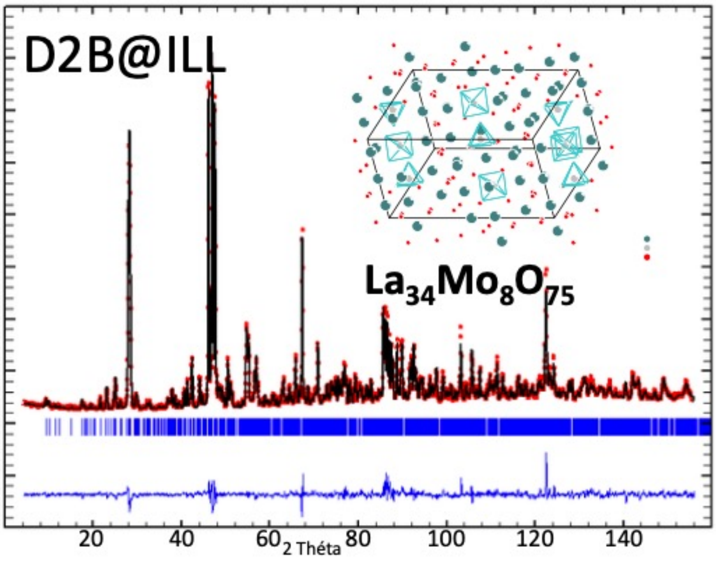
Exploring phase diagrams with neutron powder diffraction

F. Goutenoire[a], Q. Ye[a], E. Suard[b]

[a]*Institut des Molécules et Matériaux du Mans UMR6283*
[b]*Institut Laue Langevin, Grenoble*

francois.goutenoire@univ-lemans.fr

In many chemistry fields the knowledge of phase diagrams is not known or partially. In the past, our effort has focused in binary and ternary phase diagrams: La_2O_3-MoO_3, La_2O_3-WO_3 and nowadays La_2O_3-MoO_3-Nd_2O_5. Some primary information were available, mainly the composition but without crystallographic data due to the difficulty to obtain single crystal [1]. Our work consist to elucidate the structure from the powder diffraction data X-rays and neutron. Many structures has been discovered: $La_2Mo_2O_9$ [2], $La_2Mo_4O_{15}$ [3], $La_{34}Mo_8O_{75}$[4], $La_6W_2O_{15}$ [5], $La_{18}W_{10}O_{57}$[6], $La_2W_2O_9$ [7], $La_5Mo_2NbO_{16}$ [8], $La_7Mo_7O_{30}$ [9], $La_7W_4Nb_3O_{30}$[10], $La_6Mo_8O_{33}$[11] and La_3WNbO_{10}[12].



References

[1] High temperature phase relation in the system La2O3-WO3,"M. Yoshimura et al., Mat. Res. Bull 1976.
[2] Designing fast oxide-ion conductors based on La2Mo2O9, Ph. Lacorre et al, Nature 404, 856–858 (2000).
[3] F. Dubois et al., Journal of Solid State Chemistry, Vol. 159-1, 2001, 228-233.
[4] T. Dan Vu, Firas Krichen et al., Cryst. Growth Des. 2019, 19, 11, 6074–6081.
[5] M-H Chambrier et al., Journal of Solid State Chemistry, Vol. 183-6, 2010,1297-1302.
[6] M-H Chambrier et al., Inorg. Chem. 2009, 48, 14, 6566–6572.
[7] Y. Laligant, A. Le Bail, F. Goutenoire., Journal of Solid State Chemistry Vol. 159-1, 2001, 223-227.
[8] T.D Vu et al, Journal of Solid State Chemistry, Vol. 237, 2016, 411-416.
[9] F. Goutenoire, R. Retoux, E. Suard, P. Lacorre, Journal of Solid State Chemistry, Vol.142-1, 1999, 228-235.
[10] F. Goutenoire, S.Kodjikian, E.Suard, Journal of Solid State Chemistry, Vol. 178-9, 2005, 2811-2817.
[11] V. Brizé et al. Journal of Solid State Chemistry, Vol. 177-7, 2004, 2617-2627.
[12] T.D. Vu et al, Journal of Solid State Chemistry, Vol. 229, 2015, 129-134.

Fundamental and Particle Physics

ROOM

09:00-10:30

Neutrino-Nucleus Coherent Scattering, ESS Research Program Status

Francesc Monrabal Capilla[a]

[a] *Donostia International Physics Center (DIPC), Paseo Manuel Lardizabal, 4, E-20018, Donostia-San Sebastián, Spain*

monrabal18@gmail.com

The recent detection of the coherent elastic neutrino-nucleus scattering (CEvNS) opens the possibility to use neutrinos to explore physics beyond the standard model of particle physics with small size detectors. however, the CEvNS process generates signals at the few keV level, requiring the development and use of very sensitive detecting technologies for its observation. The neutrinos produced at the European Spallation Source (ESS) have been identified as the best possible alternative for the exploration of potential new physics in CEvNS. We propose to combine different detector technologies capable of observing this process with lower energy threshold and better energy resolution than current detectors. In addition, the combination of these technologies will allow for a complete phenomenological exploitation of the coherent signal. This is particularly relevant at the ESS, since, thanks to the large flux of neutrinos produced at the facility, experiments will not be limited by statistics.

In this talk I will present the two main complementary projects currently under construction: one based in the development of cryogenic csi scintillators, and the other in the use of high pressure gas TPC technology, using several noble gases, for the detection and reconstruction of the CEvNS events at the ESS.

Measurement of the Fierz interference term with PERKEO III

Karina Bernert[a], Max Lamparth[a], Hartmut Abele[b], Andreas Doblhammer[b], Erwin Jericha[b], Jens Klenke[c], Annabel Kropf[a], Kathrin Lehmann[c], Heiko Saul[a], Ulrich Schmidt[d], Torsten Soldner[e], Bastian Märkisch[a]

[a]Technical University of Munich, Garching, Germany
[b]Atominstitut, Vienna University of Technology, Vienna, Austria
[c]FRM II, Garching, Germany
[d]Heidelberg University, Heidelberg, Germany
[e]ILL, Grenoble, France

Karina.bernert@tum.de

Measurements of the free neutron decay enable a variety of tests of the Standard Model of particle physics. Observables of the decay are, among others, the beta asymmetry A, the proton asymmetry C, and the Fierz interference term b. From precision measurements of A the CKM matrix element V_{ud} may be determined, whereas measurements of C provide limits on right handed currents. A non-zero Fierz term b would signal the existence of scalar and tensor interactions beyond the Standard Model.

Determinations of these neutron decay parameters were pursued by the PERKEO III experiment by measurements of the electron and/or proton energy spectrum, during multiple runs at the ILL PF1b facility. For these measurements we used a pulsed beam of cold neutrons to control major systematic effects. This beam is guided into the decay volume of the experiment, in which some of the neutrons decay. The charged particles from the decay are then guided by a magnetic field towards one of two detectors, depending on towards which hemisphere they were directed to. The detectors are identical scintillation detectors with a PMT readout. With this measurement technique PERKEO III delivers the currently most precise values for A and b with a polarized neutron beam.

We present experimental details of the 2019/2020 campaign to measure the electron spectrum of unpolarized neutrons to extract an improved limit for the Fierz interference term b and the ongoing analysis.

Diffraction enhanced experiments for particle physics

Michael Jentschel[a], *Valery Nesvizhevsky*[a]

[a]*Institut Laue Langevin, Grenoble, France*

jentsch@ill.fr

The wave character of a neutron and according optical phenomena like diffraction and interference allow to set up very sensitive experiments in particle physics. Well-known examples are crystal diffraction based neutron interferometers probing fundamental phenomena of quantum mechanics or whispering gallery experiments where neutron wave interferences test sensitively short range interaction in the quantum regime. Other experiments make use of dynamical diffraction phenomena in perfect single crystals to form extremely low divergent neutron beams and use these properties as sensitive probe to study neutron interaction with gravity, electric fields or yet unknown interactions. In some cases the diffraction process itself – being a coherent superposition of multiple scattering events - becomes a probe for short range interaction.

We will review a number of neutron experiments in particle physics based on diffraction. We will discuss the potential of pulsed neutron beams as delivered by the ESS and compare them to opportunities at a research reactor such as the ILL.

The neutron - anti-neutron oscillation (NNBAR) experiment at the ESS

Richard Wagner[a] on behalf of the NNBAR collaboration

[a]Institute Laue-Langevin, Grenoble, France

wagnerrichard@ill.fr

Although the Standard Model (SM) of particle physics is undisputedly established and well-tested, a number of major open questions in modern physics remain. These include the observed asymmetric abundances of matter and antimatter in our known universe after baryogenesis. The NNBAR experiment [1] was proposed to search for baryon number violation (BNV) due to neutron (n) – anti-neutron (n) con- versions. A process that could explain the aforementioned asymmetry. The BNV process may occur as free neutrons propagate via ballistic motion to a detector, where anti-neutrons will annihilate and be detected via their multi-pion decay signature. A schematic of the setup of the planned experiment is shown in Fig.1.

To reach an increase in sensitivity of three orders of magnitude over the current limit, obtained at a previous experiment at the ILL [2], the experiment is to take ad- vantage of the expected unique high intensities of cold neutrons from the second, lower moderator system at the ESS. Design and optimization of the various components comprising the experiment are currently explored and developed in course of the HighNESS project [3]. This includes the design of the moderator for cold neutrons, the transport of the neutrons to the detector region and the detector itself.

We will give an overview on the present state of the work on the NNBAR experiment with special focus on the optics and detector system.

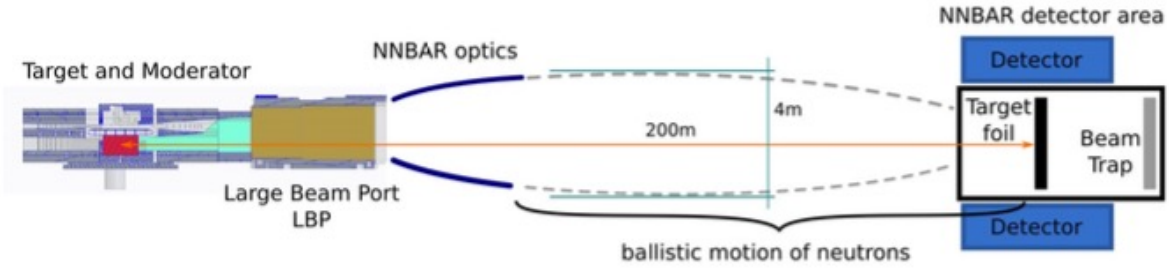


Figure 1 Schematics of the NNBAR Experiment (not in scale)

References

[1] Addazi, A., et al. "New high-sensitivity searches for neutrons converting into antineutrons and/or sterile neutrons at the HIBEAM/NNBAR experiment at the European Spallation Source." Journal of Physics G: Nuclear and Particle Physics 48.7 (2021): 070501.
[2] Baldo-Ceolin, Massimilla, et al. "A new experimental limit on neutron-antineutron oscillations." Zeitschrift für Physik C Particles and Fields 63.3 (1994): 409-416.
[3] Santoro, V., et al. "Development of a High Intensity Neutron Source at the European Spallation Source: The HighNESS project." arXiv preprint arXiv:2204.04051 (2022)

Status of the PanEDM neutron electric dipole moment experiment

Hanno Filter *[*,a] & *Tom Neulinger* [b] & *Oliver Zimmer*[*,b]

**on behalf of the PanEDM collaboration*

[a] *Technische Universität München, Garching, Germany*
[b] *Institut Laue-Langevin, Grenoble, France*

hfilter@tum.de

Neutron Electric Dipole Moment searches typically compare spin precession of trapped ultra-cold neutrons (UCN) with a stable clock, in an applied high electric field. One approach to limit systematic uncertainties in this type of experiment employs two storage chambers, allowing for simultaneous differential measurements with two electric field orientations. In PanEDM [1] this approach is supported by exceptional low-frequency magnetic shielding [2], advanced optical magnetometry systems [3], and a high-density superthermal UCN source – SuperSUN [4].

The PanEDM experiment is currently under commissioning at the Institut Laue-Langevin, and characterisations of individual systems continues in parallel with preparations for first UCN production at SuperSUN. We present an overview of the current status of the PanEDM experiment. In addition, we report on measurements carried out at SUN2, the predecessor of SuperSUN, to investigate solutions for UCN transport and storage in PanEDM.

References

[1] D. Wurm et al., EPJ Web of Conferences 219, 02006 (2019)
[2] I. Altarev et al., J. Appl. Phys., vol 117, 183903 (2015)
[3] M. Rosner et al,. Appl. Phys. Lett. 120, 161102 (2022)
[4] O. Zimmer and R. Golub, Phys. Rev. C, vol. 92, 015501 (2015).

Friday 7 October

Large Scale Structures and Hard Matter

ROOM

09:00-10:30

Topological non-collinear magnetism: from skyrmions to solitons

Grace Causer[a]

[a]*Physik-Department, Technische Universität München, 85748 Garching, Germany*

grace.causer@tum.de

The discovery of topological protected states in chiral magnets inspired a renaissance in the field of non-collinear magnetism, and led to the identification of several long-wavelength spin objects including skyrmions, merons, solitons, and chiral bobbers. Small angle neutron scattering (SANS) has provided key insights on these magnetic textures in bulk materials, whose associated topological properties are discussed in the context of low-energy information carriers. SANS of such structures in thin films or micro-structured bulk materials is, however, strongly limited by the tiny scattering volume and the prohibitively large background scattering by the substrate and support structures.

In this talk I will discuss near-surface SANS, performed slightly above the critical angle of reflection, as a route to overcome the shortcomings of transmission SANS for extremely small magnetic sample volumes in the thin-film limit. We perform proof-of-concept measurements on bulk MnSi and establish equivalent scattering patterns of the helical, conical, and skyrmion lattice phases between transmission and near-surface SANS geometries [1]. Near-surface SANS performed on epitaxial MnSi reveals a phase diagram which differs distinctly from bulk, and comprises of a single-domain, out-of-plane propagating helix with $\lambda = 11.5$ nm in zero-field. Our experimental findings are supported by micromagnetic simulations which depict a magnetic phase diagram dominated by a field-induced unwinding of soliton layers. Field and temperature history provide specific routes for the nucleation of the distinct soliton phases, comprising namely of four-, three-, two-, and single-soliton layers depending on the field strength. This work showcases the applicability of NS-SANS for the study of nano-confined magnetic materials, which would otherwise possess insufficient scattering volumes to be measured in transmission, and provides insights into the role of anisotropy and dimensionality on the phase diagrams of micro-structured bulk materials.

References

[1] G. L. Causer, et al., *J. Appl. Cryst., under review* (2022)

Analysis of time-of-flight SANS data on mesoscopic crystals such as flux line lattices

E. Campillo[a], M. Bartkowiak[b], O. Prokhnenko[b], P. Smeibidl[b], E. M. Forgan[c] and E. Blackburn[a]

[a]Division of Synchrotron Radiation Research, Lund University, SE-22100 Lund, Sweden
[b]Helmholtz-Zentrum Berlin für Materialien und Energie, Hahn-Meitner-Platz 1, D-14109 Berlin, Germany
[c]School of Physics and Astronomy, University of Birmingham, Birmingham B15 2TT, UK

emma.campillo@sljus.lu.se

Bragg diffracted intensities and *q* values for crystalline structures with long repeat distances may be obtained by small angle neutron scattering (SANS) investigations. We have developed a method and an adapted Christen formula [1] for use on TOF instruments to obtain the form factor from the integrated intensity detected in vortex lattices in superconductors [2]. We illustrate this with data obtained from high magnetic field measurements on the high-temperature superconductors $YBa_2Cu_3O_7$ [3] and $Ca_{0.15}Y_{0.85}Ba_2Cu_3O_7$ [4] at the former HFM/EXED instrument at Helmholtz-Zentrum (Berlin). As a result of this, we have shown that in $YBa_2Cu_3O_7$ and $Ca_{0.15}Y_{0.85}Ba_2Cu_3O_7$ [3,4], there are deviations from the London model at the highest fields measured (Figure 1). Our methods may have application to the study of other mesoscopic crystalline structures using SANS instruments at pulsed sources.

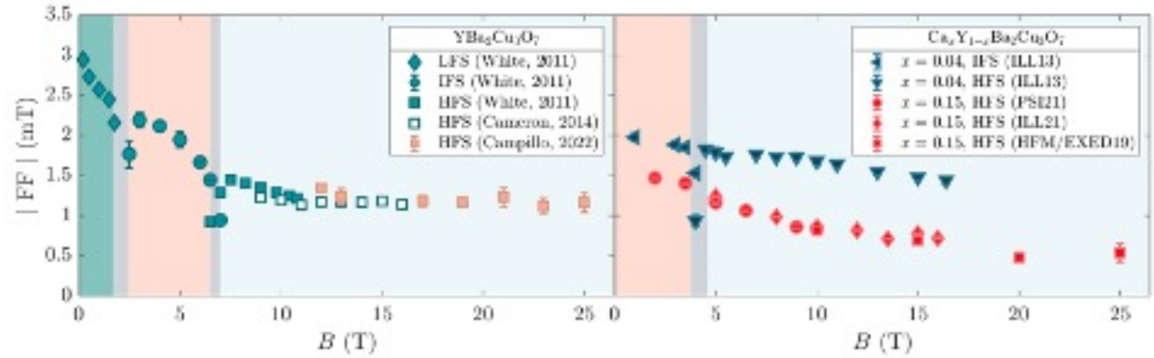


Figure 1. Evolution of the form factor with magnetic field at base temperature for (left) $YBa_2Cu_3O_7$ [3] and (right) $Ca_{0.15}Y_{0.85}Ba_2Cu_3O_7$ [4]. The different coloured regions denote different vortex lattice structures.

References

[1] D. K. Christen *et al.*, Phys. Rev. B, 15, 4506, (1977).
[2] E. Campillo, *et al.*, submitted to J. Appl. Cryst.. (2022).
[3] E. Campillo, *et al.*, Phys. Rev. B, 105, 184508 (2022).
[4] A. Cameron & E. Campillo, *et al.*, submitted to Phys. Rev. B (2022).

Vortex matter transport phenomena of the IMS

Xaver S. Brems[a,b], Sebastian Mühlbauer[a], Wilmer Y. Córdoba-Camacho[c], Arkady A. Shanenko[c,d], Alexei Vagov[e,d], Jose A. Aguiar[c], Robert Cubitt[b]

[a]Heinz Maier-Leibnitz Zentrum, Munich, Germany
[b]Institut Laue-Langevin, Grenoble, France
[c]Universidade Federal de Pernambuco, Recife, Brazil
[d]HSE University, Moscow, Russia
[e]Universität Bayreuth, Bayreuth, Germany

xaver.brems@tum.de

The elemental superconductor niobium features the intermediate mixed state (IMS), the coexistence of flux-free Meissner state and mixed state domains (fig. 1 (a)) [1]. Besides being a prominent example of superconducting vortex matter, the IMS can also act as a highly tuneable model system for universal domain physics [2]. Applying a sufficiently high current induces the flux flow state, where the vortices of the mixed state domains are de-pinned from the pinning centers and start moving orthogonal to the applied current. In contrast to the canonical mixed state, non-trivial ordering phenomena are expected in the flux flow regime of the IMS due to the highly heterogeneous domain structure.

In a recent study [3] combining transport measurements and small-angle neutron scattering (SANS) on a Nb single crystal sample we could verify the existence of the IMS in the state of flux flow. Our main result is an observation of a transition from isotropic to anisotropic IMS scattering, indicating, that the IMS rearranges itself into a stripe superstructure in the flux flow regime (fig. 1 (b,c)). The stripe pattern is aligned perpendicular to the current direction along the motion of the vortices.

We recently extended our measurement suite to include AC susceptibility. The custom-built AC susceptometer (fig. 1 (d)) can also be used during neutron experiments and in combination with the transport measurement setup. We present pre- liminary AC susceptibility results (fig. 1 (e)) highlighting the potential of a combined susceptibility, transport and neutron setup for the study of the IMS under current.

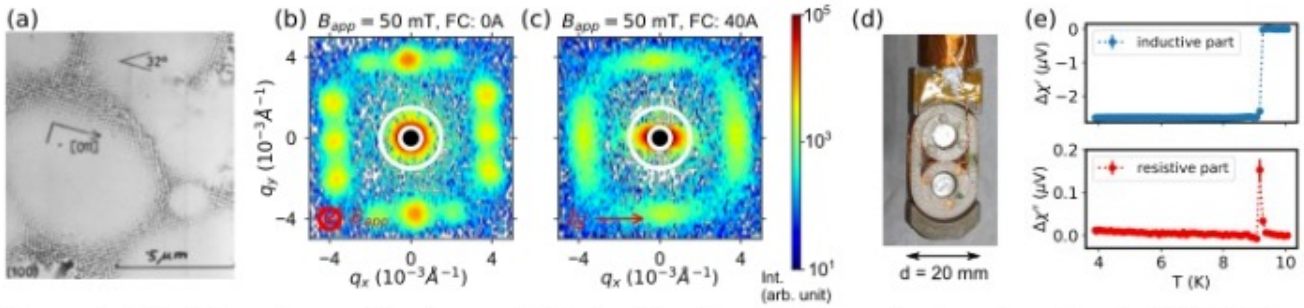


Figure 1: (a) Bitter decoration image [1] showing the IMS domain structure (b,c), 2D SANS pattern with zero current (b) and applied current I = 40 A (d), (d) Photo of the custom-built AC susceptometer, (e) Exemplary susceptibility data showing the superconducting transition temperature (Tc = 9.2K) of a small sample (4 mg weight) of pure Nb in zero field.

References

[1] Brandt and Das 2011 J. Supercond. Nov. Magn. 10.1007/s10948-010-1046-8
[2] Backs et al 2019 Phys. Rev. B 10.1103/PhysRevB.100.064503
[3] Brems et al 2021 Supercond. Sci. Technol. 10.1088/1361-6668/ac455e

In-situ small-angle neutron scattering study of hydrogen physisorption in nanoporous carbons

Sebastian Stock[a, b], Malina Jop[a], Bruno Demé[b], Viviana Cristiglio[b], Nikolaous Kostoglou[a], Oskar Paris[a]

[a]Montanuniversität, Leoben, Austria
[b]Insitute Laue-Langevin, Grenoble, France

Sebastian.Stock@unileoben.ac.at

Molecular hydrogen (H_2) is recognized as a promising substitute for carbon-rich fossil fuels, being an environmentally friendly alternative when produced with renewable energy. However, the efficient and safe storage is one of the main challenges towards wide utilization of H_2 as a fuel. Physisorption of molecular H_2 in high-surface area nanoporous materials is considered as an alternative volume-efficient solution and is associated with fast adsorption/desorption kinetics and complete reversibility due to the low binding energies/weak van der Waals forces between the solid surface and the gas molecules. Very narrow pores in the sub-nanometer range with overlapping potentials from opposite pore walls act as preferential adsorption sites for H_2. Activated carbons are considered as promising materials, as they can be produced with a high fraction of sub-nanometer pores [1]. Here, we combine in-situ small-angle neutron scattering (SANS) and gas sorption measurements using H_2 and D_2 at cryogenic temperatures. The SANS signals at different gas pressures were recorded with the high resolution diffractometer D16 at the Institute Laue-Langevin (France), which allowed to cover the scattering signals originating from a wide range of pore sizes. The strong interaction of neutrons with the hydrogen nucleus and the different interaction with D_2, opens the possibilities to probe the density of gas molecules in their confined spatial arrangement, as well as to evaluate possible isotope effects. The results of these measurements will be discussed in the framework of classical models from literature [2, 3].

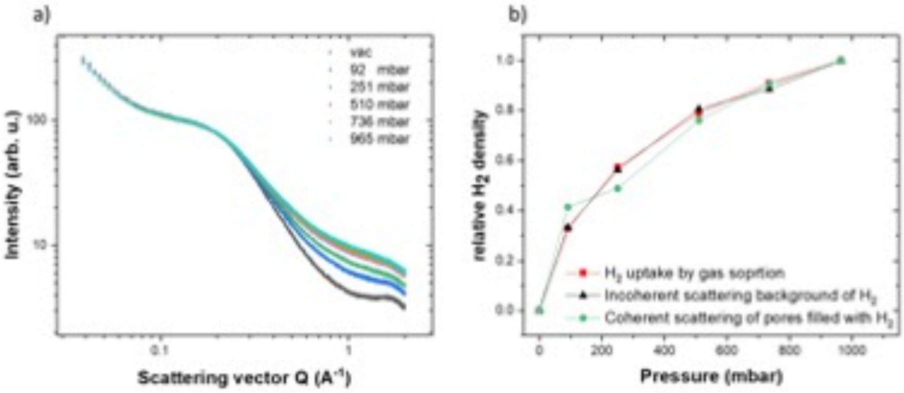


Figure 1 a) SANS signals collected at 77 K and different H_2 pressures and b) H_2 density relative to the measurement at 965 mbar obtained by different approaches (red squares: uptake measured by the gas sorption system, black triangle: density obtained from the incoherent scattering background of the H_2 molecules in the pore space and green circle: coherent scattering signal of the H_2 molecules in the pore space)

References

[1] Kostoglou, et al., Chem. Eng. J. 427 (2022) 131730. https://doi.org/10.1016/j.cej.2021.131730
[2] L. He, et al., Carbon N. Y. 80 (2014) 82–90. https://doi.org/10.1016/j.carbon.2014.08.041
[3] N.C. Gallego, et al., J. Am. Chem. Soc. 133 (2011) 13794–13797. https://doi.org/10.1021/ja202432x.

Neutrons used as a probe to discriminate hydrates from hydrocarbons (withdrawn)

S. Bouat[a], L. Pinier[b], X. Sebastian[b], A. Losko[c], R. Schütz[c], M. Schulz[c], Z. Revay[c], Z. Ilic[c,d,f], E. Mauerhofer[e], T. Brückel[e,f] and R. Gilles[c]

[a] Science-SAVED, 310 impasse Sansaret, 38 410 Grenoble, France
[b]TechnipFMC, 6/8 allée de l'Arche, 92 973 Paris La Défense, France
[c]Heinz Maier-Leibnitz Zentrum (MLZ), TU München, Lichtenbergstr. 1, 85748 Garching, Germany
[d]Jülich Centre for Neutron Science (JCNS-4) at MLZ, Forschungszentrum Jülich GmbH, Garching, Germany
[e]Jülich Centre for Neutron Science (JCNS-2), Peter Grünberg Institut (PGI-4) Forschungszentrum Jülich GmbH, Jülich, Germany
[f]RWTH Aachen University, Lehrstuhl für Experimentalphysik IVc, 52056 Aachen, Germany

contact@science-saved.com

Locating and detecting hydrate plugs inside submarine pipelines, in situ and contactless, is of first importance to get rid of them and restore pipeline flow, avoiding accumulation of new submarine pipelines deposited on the sea floor. This detection can be done using neutron activation analysis that allows to probe atoms and explore in detail the material composition. Cold and fast neutron- beam instruments at Heinz Maier-Leibnitz Zentrum were used to show that neutrons penetrate through the thick wall and the insulation of submarine pipelines and even the induced gamma radiation can be detected outside to perform a non-destructive chemical analysis within the pipe [1]. It was found that the change in the hydrogen concentration caused by a hydrate plug formation can be detected in seconds, even within quasi-real surrounding conditions (Figure 1); while with a detailed analysis at a given spot lasting for a few hours, it is possible to unambiguously identify the hydrate phase inside the hydrocarbon phase. The path has been smoothed for a possible on-board equipment, in a submarine Remote Operated Vehicle, that could "fly" above sub-sea pipelines, locating hydrate plugs *in situ* and contactless.

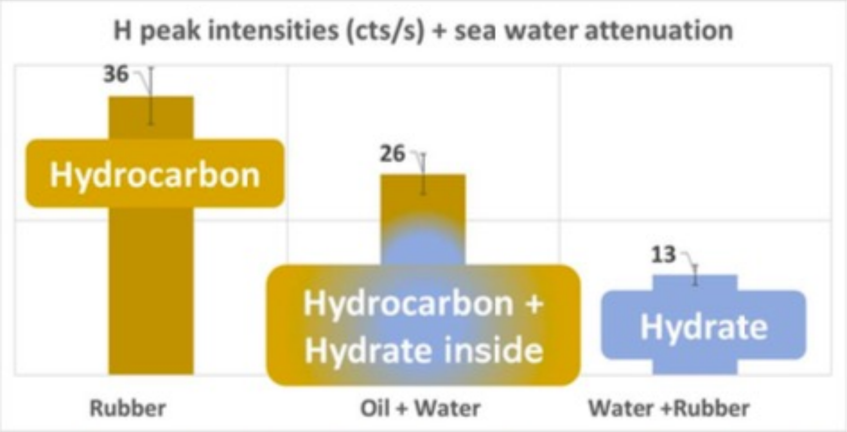


Figure 1: Hydrogen peak intensities from different sample representing hydrocarbons and hydrates, measured with salty water added in the neutron beam path to account for sea water attenuation.

References

[1] S. Bouat, L. Pinier, X. Sebastian, A. Losko, R. Schütz, M. Schulz, Z. Revay, Z. Ilic, E. Mauerhofer, T. Brückel and R. Gilles, "Detection of hydrate plugs inside submarine pipelines using neutrons", Nondestructive Testing and Evaluation (2022), Vol. 37 (3), pp. 245-257.

Investigating the superconducting state of 2H-NbS2

Ahmed Alshemi[a], Emma Campillo[a], Robert Cubitt[b], Edward M. Forgan[c], Mahmoud Abdel-Hafiez[d], Elizabeth Blackburn[a]

[a]Division of Synchrotron Research, Lund University, Lund, Sweden
[b]Institute Laue Langevin, Grenoble, France
[c]School of Physics and Astronomy, University of Birmingham, Birmingham, UK
[d]School of Physics and Astronomy, Uppsala University, Uppsala, Sweden

ahmed.alshemi@sljus.lu.se

To date, several materials have been proposed as hosts for the Fulde-Ferrell-Larkin-Ovchinnikov (FFLO) spatially modulated superconducting state [1], but direct experimental proof has been challenging, with only the organic superconductors providing uncontested evidence for the state [2]. The FFLO state is expected to develop at high fields and low temperatures in materials with strong Pauli paramagnetic effects, an anisotropic Fermi surface, and clean superconductivity.

2H-NbS2 has recently been proposed as a potential candidate material based on torque magnetometry, specific heat and thermal expansion measurements as a function of orientation in magnetic field. Transition metal dichalcogenides (TMDs) are strongly anisotropic layered super- conductors in which the two-dimensional planes are weakly coupled by van der Waals forces. The upper critical field of superconducting TMDs in the basal plane is shown to be dramatically enhanced by a special form of Ising spin orbit coupling [3]. When the field is applied exactly in the plane, the upper critical field increases dramatically above 16 T, beyond the limit expected from the Pauli paramagnetic effect. This behavior is reminiscent of the organic superconductors where the FFLO state is seen.

In order to investigate the superconducting anisotropy, effects of Pauli paramagnetism as well as possible FFLO diffraction signature, we used small angle neutron scattering (SANS) instrument to study the vortex lattice as a function of magnetic field and the angle between field and the c axis (Omega). We have successfully observed the vortex lattice in this material and the measurements have also shown strong intrinsic superconducting anisotropy between the c axis and the basal plane.

References

[1] J. Wosnitza, Annalen der Physik 530, 1700282 (2017).
[2] C. Agosta, Crystals 8, 285 (2018).
[3] C.-W. Cho et al., Nature Comm. 12,3676 (2021).

Spectroscopy and Soft Matter

ROOM

09:00-10:30

Hemoglobin S polymerization and diffusion in hemoglobin mixtures $HbF_xHbS_{(1-x)}$ and implication to Sickle cell disease

S. Longeville[a], A. Chenneviere[a], , O. Czakkel[b], B. Farago[b], L. Porcar[b], N. Hebert[c,d,e], L. Kiger[d,e], A.-L. Pham Hung d'Alexandry d'Orengiani[e] and P. Bartolucci[d,e]

[a]Laboratoire Léon Brillouin, CEA-CNRS, Université de Paris-Saclay, CEA Saclay, 91191 Gif-sur-Yvette, France
[b]Institut Laue-Langevin (ILL), 71, avenue des Martyrs - CS 20156, 38042 Grenoble cedex 9 – France
[c]Etablissement Français du Sang, Créteil, France
[d]Univ Paris-Est Créteil, INSERM, IMRB, LABEX GRex, Créteil, France
[e]UMGGR - Unité des maladies génétiques du globule rouge, Groupe Hospitalier Henri Mondor, APHP, Créteil, France

slongeville@cea.fr

Sickle cell disease (SCD) results from the polymerization of the hemoglobin found in red blood cells (RBC) under low oxygen partial pressure (pO_2). The hemoglobin S (HbS), responsible of this blood disorder, is a variant of normal human hemoglobin A0 (HbA0) with the substitution of a hydrophilic glutamic acid by the hydrophobic valine. It induces polymerization of HbS, when the protein is in its deoxygenated state (ie low pO_2) and very concentrated. Such polymerization leads to the deformation of RBCs which can cause obstruction of capillary vessels, it increases blood pressure and can ultimately lead to organs death. The stimulation of fetal hemoglobin (HbF) synthesis is a major target of interest in SCD because it leads to a hemoglobin mixture $HbF_xHbS_{(1-x)}$ in blood with HbF partially inhibiting HbS polymerization depending on its concentration.

It is thus of interest to investigate and correlate the structure and the dynamics of HbS and HbF mixtures to better understand 1- how HbF will inhibit HbS polymerization, under which HbF fraction and total hemoglobin concentration and pO_2. And 2- gain insight on the oxygen exchange process at the RBC level. In recent experiments[1], we have shown that Hb diffusion inside RBCs is similar to that in solution at the same concentration[2]. We have shown that not only the diffusion of Hb is necessary to obtain the full oxygenation of the RBC during the limited time of transit in the capillary close to the alveolar sac but the concentration of Hb inside RBCs corresponds to an optimum oxygen transport for an individual under physical activity.

We studied simultaneously the Hb polymerization process and the diffusion of free Hb remaining in solution using neutron spin-echo spectroscopy. Different mixtures of $HbF_xHbS_{(1-x)}$ (with x=0, 0.15, 0.24 and 1) were studied at pH and under different pO_2 ranging from 0 to 20%.

These studies show how the polymerization process is strongly affected by the pO_2 and the fraction of HbF present in solution (x). Moreover, we show how the free (non polymerized) Hb diffusion is affected by polymerization.

References

[1] S. Longeville and L. Stingaciu, Scientific Report, 2017, 7, 10448.
[2] W. Doster and S. Longeville, Biophys. J. (2007) **93** (4) , 1360-1368.

Looking for plastic phases in water and ammonia ices by HP-QENS

M. Rescigno[a], U. Ranieri[a], S. Klotz[b], M. M. Kozac, S. Ninet[b], F. Datchi[b], R. Gaal[d], L. E. Bove[a,b,d]

[a]*Dipartimento di Fisica, Sapienza Universita`di Roma, 00185 Roma, Italy*
[b]*Sorbonne Universite ́, UMR CNRS 7590, Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie (IMPMC), 75005 Paris, France*
[c]*Institut Laue-Langevin, 71 avenue des Martyrs, CS 20156, 38042 Grenoble cedex 9, France,*
[d]*Earth and Planetary Science Laboratory, Institute of Physics, École Polytechnique Fédérale de Lausanne, CH- 1015 Lausanne, Switzerland*

maria.rescigno@uniroma1.it

Water is, by far, one of the most fascinating and investigated substances, given its central role in a variety of fields, ranging from biology to planetary science. Neutrons studies have largely con- tributed to characterize both the water rich phase diagram and its peculiar dynamics in a broad range of thermodynamic and confinement conditions.

In the last decade, several computer simulations works [1,2] have predicted the existence of a new exotic phase of water, called “Plastic Ice”, along the melting line between liquid water and ice VII. The dynamical behavior of this phase is intermediate between that of a liquid and the one of a crystal: water molecules are held tightly in an ordered structure but freely rotate as in a liquid state. Plasticity has been observed in literature for globular shaped molecules, like methane, where the freedom to rotate in the high temperature crystal exists before the coherence of the crystal is broken and the system melts. For water, and for ammonia-water ices, plasticity is predicted to exist only in the high pressure (several GPa) crystalline phases, along the corresponding high temperatures melting lines.

Here we present QENS measurements up to 10 GPa and 580 K realized in a Paris-Edinburg Press [3,6] on the IN6-Sharp and IN5 TOF spectrometers at ILL, complemented by Raman scattering measurements in diamond anvil cells, observing the existence of these exotic plastic states both in water and in ammonia monohydrate [7,8]. A detailed S(Q,w) lineshape analysis reveals that, at difference with classical MD simulations predictions [7], water molecular rotation is not isotropic but occurs around the main symmetry axis of the crystal on a picosecond time scale.

The existence of plastic ices under the p-T conditions encountered in the interiors of icy planets and moons of our Solar System could have important consequences on the modelling of the thermal properties of these icy bodies.

References

[1] J. L. Aragones and C. Vega, Journal of Chemical Physics 130, 244504 (2009);
[2] K. Himoto, M. Matsumoto and H. Tanaka, Phys. Chem. Chem. Phys., 13, 19876–19881 (2011);
[3] L. E. Bove, S. Klotz, Th. Strässle, M. Koza, J. Teixeira, and A. M. Saitta, Phys. Rev. Lett. 111, 185901 (2013);
[4] U. Ranieri, P. Giura, F. A. Gorelli, M. Santoro, S. Klotz, Ph. Gillet, L. Paolasini, M. M. Koza, and L. E. Bove, J. Phys. Chem. B, 120, 9051-9059 (2016);
[5] U. Ranieri, M. M. Koza, W. F. Kuhs, S. Klotz, A. Falenty, Ph. Gillet, and L. E. Bove, Nature Communications, 8, 1076 (2017);
[6] U. Ranieri, S. Klotz, R. Gaal, M. M. Koza, and L. E. Bove, Nature Communications, 12, 1958 (2021);
[7] M. Rescigno, et al. submitted (2022);
[8] H. Zhang, et al submitted (2022).

Cooperative tracer chain dynamics in highly entangled polymer melts

M. Zamponi[a], M. Kruteva[b], M. Monkenbusch[b], L. Willner[b], A. Wischnewski[b], I. Hoffmann[c], D. Richter[b]

[a]Forschungszentrum Jülich GmbH, Garching, Germany
[b] Forschungszentrum Jülich GmbH, Jülich, Germany
[c]Institut Laue-Langevin, Grenoble, France

m.zamponi@fz-juelich.de

Using neutron spin echo spectroscopy the dynamics of short unentangled tracer chains in the melt of a highly entangled polymer matrix has been investigated [1]. Independent of the tracer chain length the center of mass mean square displacement (MSD) is sub-diffusive at short times and crosses over to Fickian diffusion at longer times. The obtained diffusion coefficients on the nm scale agree very well with results from macroscopic methods but the dependence on the tracer chain length strongly deviates from the Rouse expectation. The cross-over to normal diffusion for all the different tracers occurs always at the same MSD, which corresponds to the tube diameter of the entangled host. This observation cannot be understood within the standard reptation model where within the entanglement volume simple Rouse motion is assumed, but might be explained by cooperative chain motions, where the tracer chains move cooperatively with the host chains to an extent limited by the tube size.

References

[1] M.Zamponi et al., Phys.Rev.Lett. **126**, 187801 (2021).

Shining the beam on water in human cells and tissues

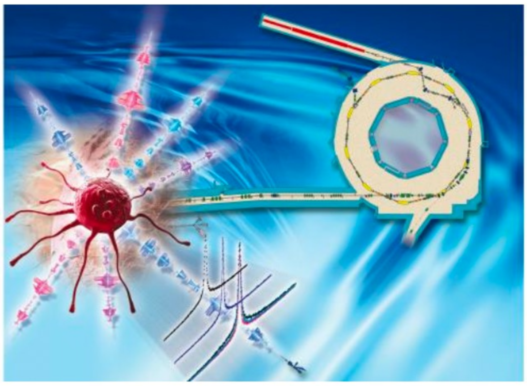
M.P.M. Marques[a,b], V. Garcia Sakai[c], I.P. Santos[a], A.L.M. Batista de Carvalho[a], A.P. Mamede[a], C.B. Martins[a], M. Sarter[c], P. Figueiredo[d], L.A.E. Batista de Carvalho[a]

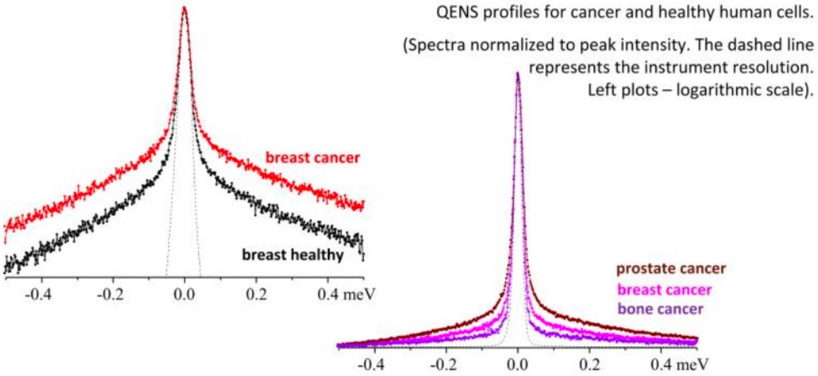
[a]Univ. Coimbra, Molecular Physical-Chemistry R&D Unit, Dep. Chemistry, Coimbra, Portugal
[b]Univ. Coimbra, Dep. Life Sciences, Coimbra, Portugal
[c]ISIS Facility, STFC Rutherford Appleton Laboratory, Chilton, Didcot, UK
[d]Portuguese Oncology Institute, Coimbra, Portugal

pmc@ci.uc.pt

Normal-to-malignant transformation in human cells is still an ill-understood process, closely related to cellular biomechanical properties. These are strongly dependent on intracellular water ́s structural and dynamical profiles, known to play a fundamental role in normal cellular activity as well as in the maintenance of the three-dimensional architecture of tissues and of the functional state of biopolymers. In this study, inelastic and quasi-elastic neutron scattering (INS and QENS) were used to: (i) probe the structural and dynamical behaviour of water confined in malignant and non-malignant human cells and tissues; (ii) monitor the impact of anticancer drugs on intracellular water (first neutron scattering study on human nucleated cells [1]). This is an innovative approach for identifying particular features associated to malignancy, and for unveiling novel pharmacological targets that may lead to an improved antitumour activity.

Changes in the dynamical behaviour of intracellular water between healthy and malignant human cells and tissues were evidenced (depending on the type of cancer), that can be considered as a promising reporter of malignancy [2-4]. These results can help to elucidate the processes under- lying carcinogenesis, invasiveness and metastatic ability, where other available methods have failed. Furthermore, the presence antineoplastic drugs was found to affect water dynamics – both within the cytoplasm and the hydration layers of biomolecules [1] – paving the way for new mechanisms of drug action, which are suggested to have a positive impact on therapeutic effectiveness.





QENS profiles for cancer and healthy human cells.

(Spectra normalized to peak intensity. The dashed line represents the instrument resolution. Left plots – logarithmic scale).

References

[1] M.P.M. Marques *et al.*, *PCCP* 19 (2017) 2702. DOI: 10.1039/C6CP05198G
[2] M.P.M. Marques *et al.*, *Struc.Dyn.* 7 (2020) 054701. DOI: 10.1063/4.0000021
[3] M.P.M. Marques *et al.*, *Int.Rev.Phys.Chem.* 39 (2020) 67. DOI: 10.1080/0144235X.2020.1700083
[4] M.P.M. Marques *et al.*, *PCCP under revision*

WASP the wide angle spin echo instrument is in user operation

Péter Falus[a], Béla Faragó[a], Peter Fouquet[a].

[a]Institut Laue-Langevin, Grenoble, France

falus@ill.fr

The first Neutron Spin Echo (NSE) instrument, IN11, was in user operation for 40 years. The newest spin echo instrument WASP took the relay and just had its first full year of user operation. I will use this occasion to review how the design of the wide angle NSE spectrometers has developed over the years. I will discuss how a WASP type instrument is a good candidate to close the 'ESS capability gap' and which new capabilities we can expect from WASP on a pulsed source.

Apart from WASP, all non-resonant Neutron Spin Echo spectrometers use the basic IN11A design where the precession field is generated by long solenoids along the neutron beam. This construction limits the angular detector coverage and count rate of the instruments. Last century there have been two attempts to make a wide-angle coverage neutron spin echo instrument. IN11C at ILL was equipped with a flattened solenoid downstream of the sample and was in use until recently. It had a 30 degree-wide angular coverage but a very limited resolution. This instrument was practically trading resolution for intensity. The SPAN instrument[1] at HZB used a pair of coils in the anti-Helmholtz configuration creating an azimuthally symmetric magnetic field, which, in theory, could allow a nearly 360 degree detector coverage. WASP uses an improved SPAN construction, and it aims to have a 500 times higher detected intensity than IN11A while the resolution remains the same.

The long construction has finished in 2018, and the instrument has seen 4 full cycles of user operation. The detailed characteristics of the instrument and the first scientific results[2][3] will be presented.

References

[1] C Pappas et al., Physica B: Condensed Matter, 283, 365-371 (2000).
[2] P Luo et al. Nature Communications, 13, 2092 (2022)
[3] H Frielinghaus et al., Frontiers in Physics, 10, 872616 (2022)

Advancing electrochemical reactor science with neutron radiography

Antoni Forner-Cuenca

Department of Chemical Engineering and Chemistry, Eindhoven University of Technology, The Netherlands

a.former.cuenca@tue.nl

Electrochemical processes are poised to play a pivotal role in the evolving global power system because the efficient interconversion of electrical and chemical energy can enable the development of green technologies that support the decarbonization of the electric grid, power the automotive fleet, and offer new opportunities for chemical manufacturing. Mass transport plays a performance-defining role in many existing and envisioned electrochemical systems. Spatial and temporal gradients in reactant concentration, influenced by the local electrode microstructure and surface properties, govern cell- and stack-level performance and durability. However, the cell and stack performance is typically characterized using traditional electrochemical diagnostics (e.g. polarization curves, electrochemical impedance spectroscopy) and the influence of various components is *macroscopically* evaluated via comparison between novel materials and the current state-of-the-art. While this is a valid approach to identify promising candidates, quantitative insights are limited as performance-limiting factors are often dependent on coupled *microscopic* processes that. *Operando* imaging has been instrumental in the advancement of polymer electrolyte fuel cells (PEFCs) and lithium-ion batteries. While a number of powerful techniques have been successful employed (e.g. x-ray tomographic microscopy, fluorescence microscopy, infrared thermography), neutron imaging offers unique advantages for visualizing electrochemical cells during operation.

In this presentation, I will discuss two modern applications of neutron imaging to advance the understanding of mass transport within electrochemical cells. First, I will discuss the application of neutron radiography to image multiphase flows in high-power PEFCs[1-2]. High-resolution neutron imaging was employed to visualize water distribution *in-situ* to assist the development of novel gas diffusion layers[3]. Then, I will describe our recent efforts to quantify local concentrations of active species and supporting electrolytes in redox flow batteries[4]. Here, we leverage the high attenuation of organic materials (i.e., high hydrogen content) in solution and, combined with isotopic labelling, we perform substrative neutron imaging to quantify the concentration of active species and supporting electrolytes.

References

[1] P. Boillat *et al.*, Curr. Opin. Electrochem. 5(1), 3-10, 2017.
[2] A. Forner-Cuenca *et al.*, *J. Electrochem. Soc.* 163(13), F1389-F1398, 2016.
[3] A. Forner-Cuenca *et al.*, *Adv. Mater.* 27(41), 6317-6322, 2015.
[4] R. R. Jacquemond *et al.*, ECS 241st Meeting, L04-2014, 2022.

Neutrons for Europe
USER MEETING
2022

Poster Abstracts in Numerical Order

Neutron Technique

Diffraction

Fundamental and Particle Physics

Imaging

Large Scale Structures

Spectroscopy

Science Area

Cultural Heritage

Energy

Engineering

Fundamental Research

Health Challenges

Industrial Applications

Life Science

Magnetism

Soft Condensed Matter

Poster Abstracts in Numerical Order

Poster Number	Title	Submitting Author
2	High Resolution Imaging using Tandem Macro Lenses	Alan Hewat
5	Protein Production Sweden (PPS) and biological deuteration	Wolfgang Knecht
7	Neutron diffraction study of Sr2FeMoO6	Vadim Sikolenko
9	Reconstitution of human dihydroorotate dehydrogenase into supported lipid bilayers using lipid-detergent micelles and liposomes: A neutron scattering study	Juan Manuel Orozco Rodriguez
10	Ionic liquid-water mixtures confined in nanoporous glasses studied with wide-angle neutron spin echo spectroscopy	Olaf Holderer
11	In-situ illumination of a sample during inelastic neutron scattering at cryogenic temperatures	Hamish Cavaye
14	Characterization and magnetic excitations in newly synthesized Tm2Ir2O7	Milan Klicpera
15	The upgraded D16 diffractometer at the ILL	Viviana Cristiglio
16	Insights into the microscopic mechanisms responsible for phase separation in a biphasic acidic solution for metallic ion extraction.	Marie Plazanet
18	Water confinement inside imogolite nanotubes	Arianna D'Angelo
24	Hydrogen Bonds in Hydroxyl-Functionalised Ionic Liquids Stabilise Cation-Networks and Lend a Hand to Molecular Components	Johanna Busch
25	Unraveling the Ground-State Structure of BaZrO3 by Neutron Scattering Experiments and First-Principles Calculations	Adrien Perrichon
26	From PEF to PBF: A computational spectroscopy study of furandicarboxylate-based polyesters	Paulo Claro
28	Small angle scattering and biophysical analysis of an extended MYC-MAX-DNA complex including the conserved MYC-MBIV region	Francesca Caporaletti
30	Structural changes during annealing of core-shell latex colloidal thin films	Stephan Roth
32	Assessing the structural dynamics of chloromethane derivatives through INS	Mariela Nolasco
34	Methanol diffusion in H-ZSM-5 zeolite catalysts as a function of loading and Si/Al ratio	Claire-Louise Woodward
35	Local and Nanoscale water behaviour in zeolites	Alexander Porter
37	Protein diffusion during non-classical crystallization processes	Christian Beck
38	NUrF: Streamlining liquid handling & data processing and analysis for SANS beamlines	Gudrun Lotze
39	Probing model lignin monomers within acidic zeolite catalysts of varying compositions	Katie Morton
41	Structural study of mouse translocator protein (mTSPO) in different amphiphilic environments.	Christelle saade
42	Structural disorder and magnetic correlations in non-Kramers Tb2Hf2O7 pyrochlore	Victor Porée
44	Structural and dynamical properties of liquid chromophores	Eric Lindgren
45	Probing topological interactions in polymers under shear	Manuchar Gvaramia
48	Bayesian refinement of diffraction data	Erik Fransson
51	Signatures for Berezinskii-Kosterlitz-Thouless critical behavior in the planar antiferromagnet BaNi2V2O8	Ekaterina Klyushina
52	Nanostructural characterization of cardiolipin-containing tethered lipid bilayers for protein incorporation	Sebastian Köhler
55	Thermal expansion modeling of mullite-type Bi2Fe4O9, Bi2Fe2Mn2O10 and Bi2Mn4O10 across magnetic phase transitions studied by neutron elastic scatterings	M. Mangir Murshed
58	Study of phase equilibria of novel deep eutectic solvents systems	Sushma P
62	Towards measuring surface dynamics with neutrons	Maximilian Wolff
63	Longitudinal Conical Magnetic Structure in Scandium doped M-type Barium hexaferrite	Surbhi Gupta
65	Investigations of boron carbide based neutron absorption layers for an early-stage superconducting neutron detector	Mette Bybjerg Brock
66	Polarized SANS and GISANS: Research and data analysis at LU/ESS	Annika Stellhorn
67	Quantum cascade laser-based infrared spectrophotometer for the combination with small angle neutron scattering for life science applications.	Seyed Mohammad Mahdi Dadfar
69	Probing Double Exchange Spin Dynamics within Valence Delocalised Magnetic Molecules using Various Spectroscopic Techniques	Rasmus Tang Christiansen
72	The Role of Intramembrane Peptide Nanoaggregates in Bacterial Fast-killing	Mingrui Liao
73	Combining SANS, Cryo-EM and computational modeling to reveal structure of Hepatitis B virus	Wojciech Potrzebowski
74	Combined INS, FT-IR and DFT evidence of H2 induced ductility in a Pt/Al2O3 catalyst	andrea piovano
77	Flux enhancement for GISANS studies using fan shaped beams	Alexey Vorobiev
78	Effect of surface charges on structure and interactions of soft colloids	Jorg Stellbrink
79	Neutron structure of Leishmania mexicana triosephosphate isomerase variant E97Q reveals a possible general acid	Esko Oksanen
80	The effect of framework composition on molecular behaviour and controlled drug release from zeolites	Alexander O'Malley
83	Tail-unsaturation Enables Fine-tuning on the Rheology of Sugar-based Surfactants	Jia-Fei Poon
84	Multimodal Tools To Resolve Assembly Mechanisms In the α –> β transition of Recombinant Cylindriform Silk	Juanita Francis
85	Magnetoelectric coupling of rare-earth orthotantalates	Xiaotian Zhang
87	Towards a Comprehensive Picture of Temperature-responsive Elastin-like Peptides	Olga Matsarskaia
89	Facilities for Macromolecular Crystallography at the HZB	Thomas Hauß
91	MARMOT – Multiplexing on Thales	Paul Steffens
92	Solution structure of FRP-ΔNTEOCPO complex in cyanobacteria	Mina Hajizadeh omaslanolya
93	Extending MIEZE spectroscopy towards thermal wavelengths	Johanna Jochum
94	Structural Study of the Hepatitis B Virus X Protein and its Interactome	alexis clavier
100	Polarized neutron diffraction to probe local magnetic anisotropy and magnetization density	Iurii Kibalin

Poster Abstracts in Numerical Order

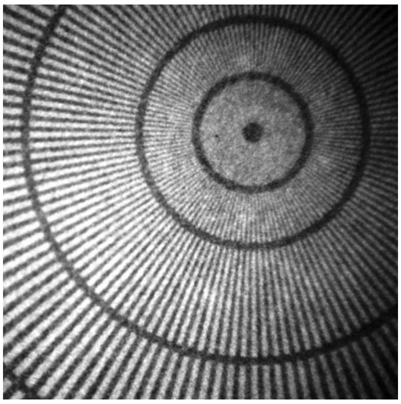
Poster Number	Title	Submitting Author
101	Protein short-time diffusion in polydisperse crowding	Tilo Seydel
102	Polyelectrolyte/Surfactant Complexes (PESCs) for Controlling Rheo-logical Behaviour – Relation between Mesoscopic Structure and Mac-roscopic Rheology	Michael Gradzielski
103	Surgical Control of Quantum Matter: Uniaxial Pressure for Neutrons	Gediminas Simutis
107	High performing computing infrastructure and use cases for neutron scattering research	Johan Hellsvik
108	Charge transfer in a redox protein – insights from neutron dynamics studies and cryo-neutron crystallography	Juliette Devos
109	The ILL Deuteration Laboratory (ILL D-Lab)	Valérie Laux
110	Neutron Scattering Studies of Organic Cation Dynamics in Low-dimensional Metal Halide Perovskites	Kanming Shi
113	Surfactant self assembly in halogen free Deep Eutectic Solvents	Elly Kim Bathke
114	On the synergy between neutron reflectometry and solid-state NMR	Ildefonso Marín Montesinos
117	Implementation of simultaneous in situ ellipsometry and infrared spectroscopy at the ESS reflectometer beamlines	Thomas Ederth
120	Nuclear structure studies at the FIPPS instrument at ILL	Giacomo Colombi
121	Chalcogenides for wearable thermoelectrics	Milos Fejercak
124	Small-Angle Neutron Scattering of a Pentameric Ligand-Gated Ion Channel Reveals a Dynamic Regulatory Domain	Marie Lycksell
125	ORSO- Open Reflectometry Standards Organisation	Thomas Arnold
126	First Experimental Observation of the Vibrational Excitations in the CDW-SC LaPt2Si2 Studied with INS	Elisabetta Nocerino
127	Exploiting symmetry with brille	Gregory Tucker
128	Spectral characterization of the SUN-2 ultracold neutron source by vertical time-of-flight	Thomas Neulinger
130	Neutron Diffraction Study of Surfactant-Skin Lipid Model Interaction	Yao Chen
131	Degradation of lipid films upon nanoparticle contact studied by simultaneous neutron reflectometry, infrared spectroscopy and ellipsometry	Reiner Dahint
133	Hardening of phonons in SrTiO3 in electric fields measured with neu-tron scattering	Henrik Jacobsen
135	Searches for Baryon Number Violation Using HIBEAM	Matthias Holl
136	Gradual emergence of superconductivity in underdoped La_{2-x}Sr_xCuO_4	Kim Lefmann
137	Liposomes under the influence of short-chain alcohols	Fernanda Alvarado Galindo
138	Investigation of candidate samples for a Bragg edge neutron imaging round robin	Ranggi Sahmura Ramadhan
139	Diffusion in supramolecular gels for drug delivery	Riccardo Morbidini
140	Evolution of magnetic stripes under uniaxial stress in La_(1.885) Ba_(0.115) CuO_4 studied by neutron scattering	Kristine Krighaar

Poster Number	Title	Submitting Author
141	Suppression of magnetic order and enhancement of magnetic fluctuations in frustrated magnets from the family of iron jarosites	Peiyuan Liu
143	Magnetic structure and spin flip transition of MnSb4Te7	Siobhan Tobin
145	Disposition of small molecules in stacked bilayers of stratum corneum lipids by neutron diffraction and selective deuteration	Christopher Garvey
147	Deuterated Clathrate Hydrates as Novel Moderator Material for a Source of Very Cold Neutrons	Valentin CZAMLER
148	Dynamics of the critical phonon modes in quantum paraelectric SrTiO3	Shiyu Deng
149	Unveiling Oxidation and Spin State of Fe in Li1-xZnxFeO2	Priyanka Priyanka
150	Molecular origins of piezoelectricity in fibroin	Christopher Garvey
151	Structural Characterisation of Nanoparticle-Supported Lipid Bilayers by Grazing Incidence X-ray and Neutron Scattering	Nico Paracini
152	Interaction of colloids with cellulose nanofibrils in aqueous solution	Constantin Harder
153	Symmetry routes towards orbital molecules in hexagonal perovskites	Struan Simpson
156	Nested mirror optics – towards efficient neutron delivery from compact high-brilliance sources	Christoph Herb
159	Effects of Layer Thickness Ratio on Crystalline Quality of CrBx/TiBy Superlattice Neutron Mirrors Grown on 4H-SiC(0001) and Al2O3(0001)	Samira Dorri
160	Correlation of Superconducting and Morphological Properties Via Polarized Neutron Tomography	Ugwumsinachi Oji
162	Investigating the superconducting state of 2H-NbS2	Ahmed Alshemi
164	Research and tuning of Y-type hexaferrites	Oskar Stepancic
165	Does oxidative damage enhance or hinder peptide insertion into lipid monolayers?	Beatrice Barletti
166	Simplifying diffraction data analysis with EasyDiffraction	Andrew Sazonov
167	Understanding the magnetic dynamics of the strongly frustrated magnet Gd3Ga5O12 using neutron scattering and the Langevin simulation program CLaSSiC	Kim Lefmann
168	Symmetry protected 1D chains in mixed-valence iron oxides	Denis Vasiukov
172	Magnetism in the distorted Kagome-lattice intermetallic RAgGe family	Camilla Larsen
173	Particle size polydispersity analysis based on a unified exponential/power-law approach to small-angle neutron scattering	Oleksandr Tomchuk
174	Kitchen-Based Light Tomography – an educational scanner for train-ing the next generation of neutron and X-ray tomography users	Emanuel Larsson
176	Probing the effect of ligand non-innocence on magnetic excitations in pyrazine-based metal-organic framework compounds	Niels Bech Christensen
177	YMIR: A controls integration neutron-free instrument	Stefanos Athanasopoulos
178	Surfactant-free microemulsions : how molecular dynamic reflects nano-structuration	Firoz Malayil Kalathil
179	Making neutron characterization tools available to industry	Hanna Leemreize
181	First results with PASTIS3 at IN20	Ursula Bengaard Hansen
182	A hitchhikers guide to the easyScience galaxy	Simon Ward
184	Amorphous proteins in weakly hydrated environments: simulation vs. experiment	Mark Telling
185	Unique opportunities for particle physics at the ESS	Albert Young
186	Henmilite: A quantum spin liquid candidate	Ankit Labh
187	Human transthyretin and amyloidosis	Trevor Forsyth
188	The upgrade of the thermal neutron TAS at ILL: from IN8 to THERMES	Andrea Piovano
189	Foam aging under free drainage analysed using associated operando techniques	Mjulien Lamolinairie

High resolution imaging using tandem macro lenses

Alan Hewat[a]
NeutronOptics, Grenoble, France
alan.hewat@neutronoptics.com

An ordinary macro camera can achieve 1:1 optical magnification. which corresponds to micron resolution with a modern CMOS or CCD detector. The problem is that very thin scintillators are needed, with low light output, the small pixels also collect little light, and macro lenses have apertures of at best f/2.8, x4 less bright than f/1.2 lens. The latter problem has been addressed [1] by using synchrotron inspired "infinity corrected optics" with 50-year old Rodenstock lenses, which are difficult to obtain and focus. An alternative "neutron microscope" [2] is only available custom made at great expense. We wanted a design that could be made with readily available commercial lenses, to satisfy most needs for imaging down to 10-20 microns, without trying to compete with the 5 micron unique-in-the-world instrument. With SR, or micro-focus x-rays, higher resolution is possible using divergent beam magnification, not available with neutrons.



The image left shows a pair of Nikkor 50mm f/1.2 lenses, connected front-to-front via a 90-degree mirror. The image on the right was obtained on the ILL NeXT D50 beamline using a 10µ thick Gd2O2S:Tb/6LiF PSI/RC-TriTec scintillator. The inner circle shows that at least **25µ resolution was obtained for an exposure of only 3s** with a neutron flux ~5x10**7 n/cm2/s [3].

A Tandem Macro camera consists of two lenses face to face, with the object at the focal point of the first lens and the image at the focal point of the second with both lenses focussed at infinity. It is claimed by PCO [4] to be comparable in efficiency to direct fibre-optic coupling to the detector, without the granularity of the fibres. The first commercial example of our tandem macro-camera is being used to develop a new type of nano-guide polymer scintillator using Transverse Anderson localization [6], with much higher resolution than possible with normal optical fibres.

References

[1] S.H. Williams, A. Hilger, N. Kardjilov et al. (2012) Journal of Instrumentation 7, P02014
[2] P Trtik, J Hovind, Ch Gruenzweig... E. Lehmann et al. (2012) Physics Procedia, 69, 169
[3] With A. Tengattini and L. Helfen (2020) on the ILL NeXT D50 beamline https://next-grenoble.fr/
[4] W. Tutsch PCO Di-cam Series white paper (2019) p.10. "Comparison of optical coupling methods"
[5] Manuel: http://neutronoptics.com/downloads/Manual-NeutronOptics-Tandem-Macro-Camera.pdf
[6] S Karbasi, C Mirr, PG Yarandi, R Frazier, K Koch, and A Mafi (2012) Opt. Lett. 37, 2304-2306

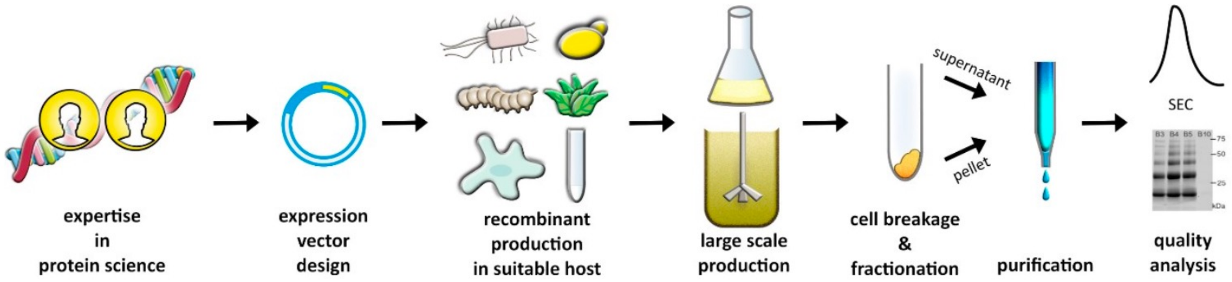
Protein Production Sweden (PPS) and biological deuteration

Malin Bäckström[a], Kristina Hedfalk[a], Sophia Hober[b], Göran Karlsson[a], Wolfgang Knecht[c], Tomas Nyman[d], Uwe Sauer[e], Hanna Tegel[b]

[a] University of Gothenburg, Box 413, 405 30 Gothenburg, Sweden
[b]Royal Institute of Technology, Stockholm, Sweden
[c]Lund University, Sölvegatan 35, Lund, Sweden
[d]Karolinska Institutet, Solnavägen9, 171 77 Stockholm, Sweden
[e]Umeå University, Linnaeus väg 10, 901 87 Umeå, Sweden

wolfgang.knecht@biol.lu.se

Protein Production Sweden (PPS; www.gu.se/pps) is a new national research infrastructure established in 2022 and focused on the production and purification of protein reagents for primarily Swedish researchers, both from academia and commercial entities. Well-established protein production platforms from five universities (the University of Gothenburg (host), Karolinska Institutet, KTH Royal Institute of Technology, Lund University, and Umeå University) collaborate to form the infrastructure and offer expert competence in 4 geographically distributed nodes (Gothenburg, Lund, Stockholm, Umeå). Researchers across Sweden can get access via a joint entry-point and have the possibility to get support based on their research needs throughout the whole process of protein production and purification, starting from project counselling and design to quality control, or for any single/multiple step(s) in the process according to the scheme below.



PPS will allow protein expression in 6 different expression systems (*E. coli*, *P. Pastoris*, Insect cells (BEVS), Plant cells, Mammalian Cells and Cell free expression).

It will also offer two Gateway modules, with one aiming at producing speciality reagents for use in X-ray crystallography. The other one aims at producing speciality reagents for and offer guidance to their use in neutron scattering experiments. (Per)deuteration of proteins is essential for neutron techniques such as neutron protein crystallography, neutron reflectometry and small angle neutron scattering. This module is thereby aiming to enable non-experts in protein production to get recombinant proteins fit for neutron scattering experiments.

For more information, please see: www.gu.se/pps

Abstract: 007

Neutron diffraction study of Sr_2FeMoO_6

V.Sikolenko[a], *S.Savvin*[b]

[a]*Karlsruhe Institute of Technology, Karlsruhe,Germany*
[b]*Institute Laue Langevin, Grenoble, France*

vadim.sikolenko@kit.edu

Double perovskite ceramic Sr_2FeMoO_6 with different amount of antisite disordering has been studied by neutron diffraction at a large temperature region 2 - 550 K using high resolution diffractometer D2B. (Proposal 5-31-2691)

The magnetic structure has been found as ferromagnetic with magnetic moments located in (bc) plane and parallel to [0 1 1] direction. Superstructural ordering of Fe^{3+} and Mo^{5+} cations has been also found.

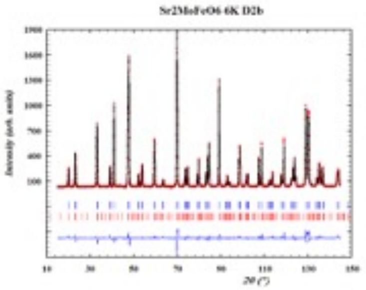


Fig.1 Neutron diffraction in Sr_2MoFeO_6 at 2K. Crystal structure, magnetic structure, experimental points, calculated spectrum and their difference are shown.

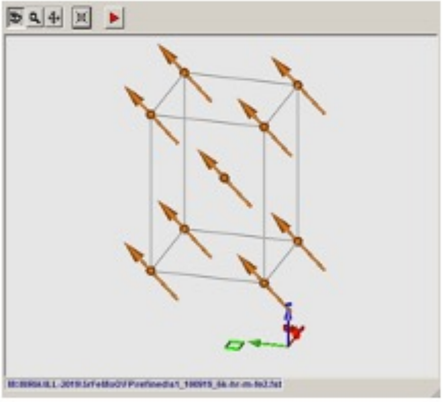


Fig.2 Magnetic structure at 2 K. Magnetic ions are iron.

Abstract: 009

Reconstitution of human dihydroorotate dehydrogenase into supported lipid bilayers using lipid-detergent micelles and liposomes: A neutron scattering study

Juan Manuel Orozoco Rodriguez[a], *Hanna P.Wacklin-Knecht*[b,c], *Luke A. Clifton*[d], *Oliver Bogojevik*[c,g],
Anna Leung[c], *Samantha Micciulla*[e], *Giovanna Fragneto*[e], *and Wolfgang Knecht*[a]

[a]*Department of Biology and LP3, Lund University, Sölvegatan 35, 223 62 Lund, Sweden*
[b]*Department of Chemistry, Lund University, Naturevetarvägen 26, 222 41 Lund, Sweden*
[c]*European Spallation Source ERIC, Box 176, 221 00 Lund, Sweden*
[d]*ISIS Pulsed Neutron and Muon Source, Rutherford Appleton Laboratory, Didcot OX11 0QX, UK*
[e]*Institut Laue-Langevun, 71 Avenue des Martys, BP 156, 38042 Grenoble, France*
[g]*Current address: Department of Biological and Chemical Engineering, Faculty of Technical Sciences, Aarhus University, Gustav Wieds Vej 10, 8000 Aarhus C, Denmark*

manuel.orozco@biol.lu.se; hanna.wacklin@esss.se

Human dihydroorotate dehydrogenase (H_sDHODH) is an enzyme anchored to the inner mitochondrial membrane (IMM) by a single transmembrane helix that catalyzes the oxidation of dihydroorotic acid to orotic acid with the simultaneous reduction of ubiquinone Q_{10} (coenzyme Q_{10}). Our aim is to investigate the interaction of H_sDHODH and ubiquinone under non-crystalline, physiologically relevant conditions using neutron reflectometry. The first step to achieve this goal is the reconstitution of H_sDHODH into supported lipid bilayers. This process still remains far from being trivial. In this study, we have therefore systematically attempted the reconstitution of H_sDHODH into supported lipid bilayers at the Si-SiO_2 water interface and analyzed the process by means of specular neutron reflection.

Three different reconstitution methods were investigated: co-adsorption of the enzyme in mixed detergent-lipid micelles containing ß-D-dodecyl maltoside (DMM), fusion of proteoliposomes, and two hybrid approaches combining the adsorption of the enzyme from protein-detergent micelles and lipid vesicle fusion. The co-adsorption of the enzyme from detergent-lipid micelles resulted in supported bilayers with low surface coverage, residual detergent, and low protein content, whereas the proteoliposome fusion yielded good lipid bilayer coverage but low protein content. On the other hand, the hybrid approach resulted in a higher protein incorporation, but also generated an additional floating layer.

Our results provide, for the first time, useful insights into the process of membrane protein reconstitution from lipid-detergent micelles in terms of the structures formed, the surface coverage, the protein incorporation, and the membrane composition achieved depending on the method used. Furthermore, the results obtained can be used to guide and inform the reconstitution of other proteins that are structurally similar to H_sDHODH.

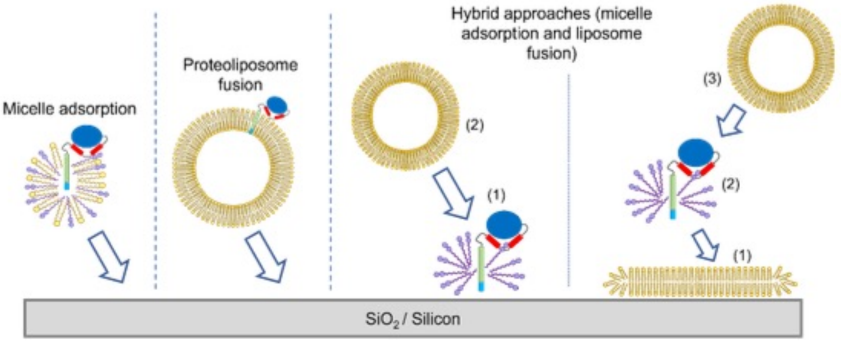


Fig. 1. Summary of the methods attempted to reconstitute human dihydroorotate dehydrogenase into supported lipid bilayers.

Ionic liquid-water mixtures confined in nanoporous glasses studied with wide-angle neutron spin echo spectroscopy

Olaf Holderer[a], *Henrich Frielinghaus*[a], *Margarita Fomina*[a],*Dominic Hayward*[a], *Purushottam S. Dubey*[a], *Sebastian Jaksch*[a], *Peter Falus*[b], *Peter Fouquet*[b], *Lisa Fruhner*[c]

[a]*Forschungszentrum Jülich GmbH, JCNS at MLZ, Garching, Germany*
[b]*Institut Laue-Langevin (ILL), Grenoble, France*
[c]*Forschungszentrum Jülich GmbH, JCNS-1 and IBI-8, Jülich, Germany*

o.holderer@fz-juelich.de

High resolution neutron spectroscopy has been used to study the dynamics of a mixture of the ionic liquid EmimAc and water, confined in a nanoporous glass [1]. Additional structural information provided by X-ray diffraction gave insight into the lamellar ordering inside the pores and the role of water in the studied case of the addition of small amounts of water. Selective deuteration of the ionic liquid further helped to elucidate the mobility of the different ions and the activation energies. Two pore sizes of the porous glass indicated the role of confinement. The new wide angle spin echo spectrometer WASP has been used to study the dynamics on a wide Q-range, where part is dominated by incoherent scattering and parts at larger Q show coherent scattering. The results are discussed in conjunction with previous backscattering spectroscopy measurements [2] on the pure ionic liquid in bulk and confinement. The confinement by the porous glass induces some distortion of domains, allowing for a higher mobility for the acetate/water compound.

References

[1] H.Frielinghaus,M.Fomina,D.Hayward,P.S.Dubey,S.Jaksch,P.Falus,P.Fouquet,L.Fruhner, and O. Holderer, *Front. Phys.* 10, 2022, 872616, doi: 10.3389/fphy.2022.872616

[2] D.Noferini,O.Holderer,H.Frielinghaus,*Phys.Chem.Chem.Phys*,22,2020,9046-9052

In situ Illumination of a sample during inelastic neutron scattering at cryogenic temperatures

Hamish Cavaye[a], Maksim Schastny[a]

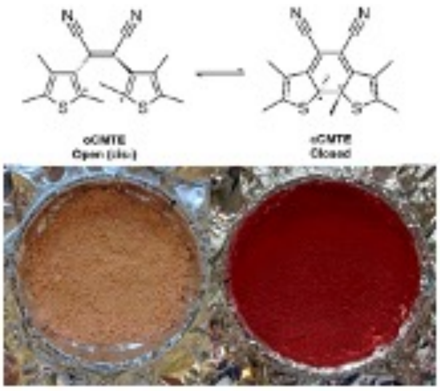
[a]ISIS Neutron and Muon Source, Rutherford Appleton Laboratory, Harwell Oxford, Didcot, England

hamish.cavaye@stfc.ac.uk

The interaction of light with matter is of great importance to a wide variety of scientific fields. Whether it is for solar energy conversion or storage, light-emitting materials, photosensitive compounds, or light-activated reactions; being able to study compounds as they are illuminated is a critically useful tool.

Photoisomerisation is a form of isomerisation that is induced by photoexcitation [1]. Photochromic materials undergo a reversible photoisomerisation between two forms, each having a different absorption spectrum, leading to a change in colour [2]. One vital aspect for characterisation of photoisomeric materials is the study of their vibrational properties, which have been linked to the mechanism for the isomerisations that they undergo [3]. Inelastic neutron scattering (INS) offers an important route to information that is highly complementary to the usual techniques of infrared and Raman spectroscopies. However, INS is often performed at cryogenic temperatures, which introduces a challenge to in-situ illumination during an INS measurement.

This work introduces a new capability developed at ISIS, to allow in-situ illumination inside a closed-cycle refrigerator (CCR), during a neutron scattering experiment. We use a well-studied photochromic material cis-1,2-dicyano-1,2-bis(2,4,5-trimethyl-3-thienyl)ethene (CMTE) to commission the new equipment and combine the results with density functional theory (DFT) simulated INS spectra to confirm the results [4].



References

[1] S. E. Braslavsky, Pure Appl. Chem., 2007, 79, 293–465

[2] H. Bouas-Laurent and H. Du¨rr, Pure Appl. Chem., 2001, 73, 639–665

[3] M. Irie and M. Mohri, J. Org. Chem., 1988, 53, 803–808

[4] H. Cavaye and M. Schastny, Phys. Chem. Chem. Phys., 2021, 23, 22324-22329

Abstract: 014

Characterization and magnetic excitations in newly synthesized $Tm_2Ir_2O_7$

Milan Klicpera[a], Kristina Vlášková[1], Daniel Staško[a], Inés Puente Orench[b,c], Tatiana Guidi[d], Martin Diviš[a]

[a]*Charles University, Faculty of Mathematics and Physics, Department of Condensed Matter Physics, Ke Karlovu 5, 121 16 Prague 2, Czech Republic,*
[b] *Institut Laue-Langevin, 71 avenue des Martyrs, CS 20156, 38042 Grenoble, France,*
[c] *Instituto de NanoCiencia y Materiales de Aragón, Pedro Cerbuna 12, 50009 Zaragoza, Spain,*
[d] *ISIS Facility, Rutherford Appleton Laboratory, Didcot, Oxon OX11 0QX, United Kingdom*

mi.klicpera@seznam.cz

$A_2Ir_2O_7$ iridates with *A* representing the rare-earth element, the subject of our continuing work, crystallize in a so-called pyrochlore structure (space group *Fd-3m*, no. 227) [1]. The crystallographic lattice consists of individual *A* and Ir nets of interpenetrating corner-sharing tetrahedra and oxygen 8- and 6-coordinate cages around the respective cations. The geometrically frustrated lattice significantly affects the magnetic properties/ground state of pyrochlores. The iridium sublattice orders magnetically, below ordering temperature T_{Ir}, in the majority of members [2]. The so-called all-in-all-out (AIAO) order has been reported for several *A*-members, e.g. [3]. Magnetic correlations between *A* magnetic moments on a frustrated lattice in an exchange interaction of Ir lead to more complex states, including spin-ice and spin-liquid states, e.g. [4,5]. In addition to the long-range Ir order, the induced order of magnetic *A* ions, and the *A*-*A* magnetic correlations, the low-temperature behaviour of $A_2Ir_2O_7$ is defined by the *A* single-ion properties, the crystal field acting on the *A* cation.

Recently, we synthesized and thoroughly characterized the previously unreported $Tm_2Ir_2O_7$ member of the family [2]. Experimental data allowed us to update and complete the $A_2Ir_2O_7$ phase diagram of T_{Ir} [2,4]. A weak magnetic signal consistent with the AIAO order of Ir sublattice was traced out in low-temperature neutron diffraction patterns. Nondispersive magnetic excitations in energy-momentum space were observed in inelastic neutron scattering spectra of $Tm_2Ir_2O_7$. The crystal field scheme and parameters of the oxide were refined and discussed with respect to the bulk properties, and supported by the microscopic Hamiltonian calculations.

References

[1] J.S. Gardner *et al.*, Rev. Modern Phys. **82**, 53 (2010).
[2] M. Klicpera *et al.*, J. Phys. Chem. C **124**, 20367-20376 (2020).
[3] H. Jacobsen *et al.*, Phys. Rev. B **101**, 104404 (2020).
[4] W. Witczak-Krempa and Y.B. Kim, Phys. Rev. B **85**, 045124 (2012).
[5] E. Lefrancois *et al.*, Nat. Commun. **8**, 209 (2017).

Abstract: 015

The upgraded D16 diffractometer at the ILL

Viviana Cristiglio and *Bruno Demé*

Institut Laue Langevin, 6, rue Jules Horowitz 38042 Grenoble Cedex 9, France

cristiglio@ill.fr

D16 is one of ILL's 2-axis cold neutron diffractometers. In terms of Q-space and Q-resolution, D16 nicely covers a wide range in q-space from that of the small angle instruments to classical high resolution diffractometers. Because of its special characteristics, D16 remains unequalled for the study of a wide range of systems in biology, physics and material science.

Its specifications have been optimized for the study of structures with relatively large periodicities of about 5 nm. These include large unit-cell lamellar organizations such as model and biological membranes or clays, two-dimensional membrane and surface lattice structures, colloidal and liquid structures, magnetic systems with large fluctuations giving rise to satellite reflections very close to Bragg peaks or small-angle scattering.

The HOPG monochromator offers a variable vertical focussing combined to vertical slit or pinhole geometry which allows for working in reflection or transmission mode. The scattering geometry obtained with large, vertically oriented samples, profits the most from the large vertical cross section of the beam at the sample position. A high resolution SANS setup is used routinely in experiments requiring the 1% wavelength bandwidth and the high angular resolution of the instrument.

In this presentation the ENDURANCE II detector project will be presented as well as examples where a major gain will be provided by the new large banana detector (protein arrangement at nm scale, nanoporous materials, semi-crystalline polymers, time resolved and/or small sample experiments such as samples in levitation, samples under mechanical stress).

The new design, the major characteristics, performances and new directions of the instrument will be presented, as well as recent examples of research performed on D16.

Insights into the microscopic mechanisms responsible for phase separation in a biphasic acidic solution for metallic ion extraction

Gautier Meyer[a], R. Schweins[b], J.-F. Dufrêche[c], Isabelle Billard[d] and Marie Plazanet[a]

[a]LIPhy, CNRS & Univ Grenoble-Alpes, Grenoble, France
[b]Institut Laue Langevin, Grenoble, France
[c]ICSM, CEA, CNRS & Univ. Montpellier, France
[d]LEPMI, CNRS & Univ Grenoble-Alpes, Grenoble, France.

marie.plazanet@univ-grenoble-alpes.fr

Ionic-liquid-based acidic aqueous biphasic solutions (AcABSs) recently offered a breakthrough in the field of metal recycling. The particular mixture of tributyltetradecylphosphonium chloride ($[P_{4,4,4,14}]Cl$), acid, and water presents the unusual characteristic of a lower solution critical temperature (LCST), leading to phase separation upon a temperature rise of typically a few tens of degrees. Here, we dig into the mechanisms driving the counter intuitive phase separation upon temperature rise observe in this system. Using small angle neutron scattering, we could identify the micellar structure under various conditions of acid and temperature, observing the micelle aggregation eventually leading to the phase separation. Temperature rise and addition of acid both induce the phase separation in an apparently similar mechanism.

Fine titration of the chloride ions present in the solution proved the exothermic adsorption of these ions at the micelle surface, increasing with temperature. Such ionic adsorption, similarly to acid addition, induces a screening of electrostatic interactions between the micelles, indirectly driving the phase separation. Such a mechanism of ionic or molecular adsorption at the micelle surface could be generalised for numbers of molecular systems presenting a Lower Critical Solution Temperature.

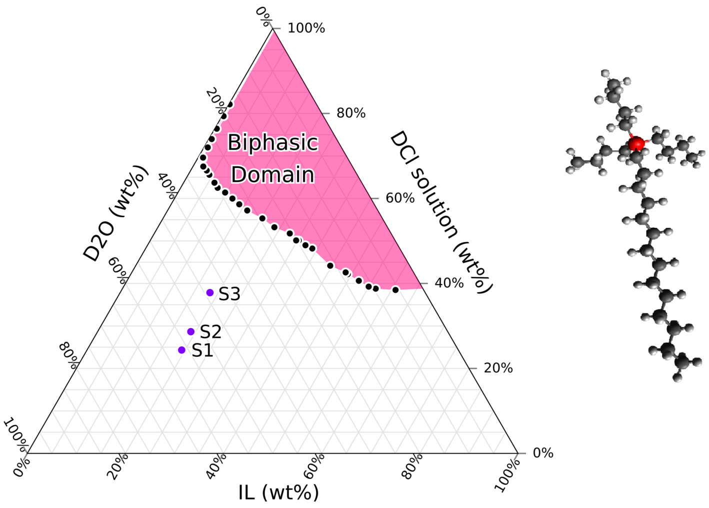


Figure: Ternary phase diagram of the $[P_{4,4,4,14}]Cl$, water and chlorhydric acid solution, and sketch of the ionic liquid cation. The phase diagram is given for deuterated acid and water for the need of neutron scattering, used for the investigation of the S_1, S_2 and S_3 solutions

References

[1] G. Meyer, R. Schweins, J.-F. Dufrêche, I. Billard and M. Plazanet, How Temperature Rise Can Induce Phase Separation in Aqueous Biphasic Solutions, J. Phys. Chem. Let. (2022), 13, 2731−2736.

Water confinement inside imogolite nanotubes

Arianna D'Angelo[a,b], *Pascale Launois*[a], *Erwan Paineau*[a], *Stéphane Rols*[b]

[a]*Laboratoire de Physique des Solides, CNRS-Unversite´Paris-Saclay, Orsay, France*
[b]*Institut Laue-Langevin, Grenoble, France*

arianna.dangelo@universite-paris-saclay.fr

Imogolite nanotubes (INT) with stoichiometry $(OH)_3Al_2O3Si(OH)$ are peculiar clay nanomaterials formed of a rolled gibbsite sheet with isolated $SiO_3(OH)$ units inside [1]. They can be synthesized by soft chemistry and Si atoms (Si-INT) can be replaced by Ge ones (Ge-INTs). The inner diameters of these hydrophilic nanotubes are truly nanometric (1.5 nm for Si-INT, 3 nm for Ge-INT). Surface properties thus prevail and INTs are model systems to study the role of interfaces on water behavior. Recent investigations on Ge-INTs [2] highlighted the stabilization of an original wetting-layer with local triangular symmetry, solid-like up to 300K.

Our study focuses on Si-INTs and on the impact of the smaller tube size on the process of water adsorption and diffusion inside their internal cavity. The results presented here were obtained by quasielastic and inelastic neutron scattering experiments, combined with Molecular Dynamics (MD) simulations using a pre-existent parametrization for the interaction potential of the imogolite [3]. The simulated QENS and Density of States of the absorbed water will be first compared to their equivalent in the case of Ge-INTs, and secondly to experimental data collected on the IN6-SHARP and PANTHER spectrometers at the ILL. The comparison with MD simulations will allow us to fit and interpret data in terms of two dynamically different populations of water.

References

[1] E. Paineau and P. Launois, Elsevier, 257-284 (2019)
[2] G. Monet et al., Nanoscale Advances, 2, 1869-1877 (2020) [3] L. Scalfi, et al., Langmuir, 34, 6748−6756 (2018)

Hydrogen bonds in hydroxyl-functionalised ionic liquids stabilise cation-networks and lend a hand to molecular components

Johanna Busch[a], *Tristan G. A. Youngs*[b], *Dietmar Paschek*[a], *Ralf Ludwig*[a,c]

[a] *Institut für Chemie, Physikalische und Theoretische Chemie, Universität Rostock, Albert-Einstein- Str. 27, 18059 Rostock,*
[b]*ISIS Faculty, STFC, Rutherford Appleton Laboratory, Didcot OX11 0QX, United Kingdom,* [c]*Department Life, Light & Matter, Universität Rostock, Albert-Einstein-Str. 25, 18059 Rostock, Germany*

johanna.busch@uni-rostock.de

Functionalisation is a novel way of adding specific chemical interactions to Ionic Liquids (ILs), thus offering the possibility to fine-tune their physical and chemical properties. In particular, hydroxyl- functionalised cations allow to anchor molecular and ionic components with hydrogen bond (HB) donating and accepting capabilities to the cations. Moreover, the cations themselves may form hydrogen-bonded contacts, thus creating cation-clusters, which are stabilised via localised HB networks [1-2]. Like-charged attraction seems to be counterintuitive at first glance, however, by using a variety of spectroscopic techniques, we could provide evidence for their existence [1-2]. Those networks are apparently stable due the fact that cation-cation HBs appear to be stronger than their cation-anion counterparts. From neutron diffraction experiments and molecular dynamics (MD) simulations the double-faced nature of HBs in hydroxyl-functionalised ILs could be revealed and their structure characterised [3-4]. By using mixtures of the pyridinium-based IL [HOC4Py][NTf2] with their non-functionalised counterpart [C5Py][NTf2], the formation and size of cation-cation clusters can even be controlled. Neutron scattering experiments and MD simulations allow insights into the formation of those cation clusters, and the effects of "hydroxyl defects" on the clusters' structure and distribution could be provided. Contrary to common expectations, ILs can be quite apolar, in particular ILs containing the [NTf2]- anion. Therefore, another important feature of the hydroxyl-functionalised IL is its provision of an HB-anchor to molecular components such as DMSO (HB acceptor only) or water (both, HB-donor and HB-acceptor), thus being able to stabilise mixtures while controlling the nano-scale structure of mixtures of functionalised ILs with their molecular co-constituents.

References

[1] A. Knorr et al., *ChemPhysChem* **2016**, *17*, 458
[2] A. Strate, T. Niemann, R. Ludwig, *Phys. Chem. Chem. Phys.* **2017**, *19*, 18854
[3] T. Niemann et al., *Angew. Chem. Int. Ed.* **2019**, *58*, 12887
[4] J. Neumann, R. Ludwig, D. Paschek, *J. Phys. Chem.* **2021**, *15*, 4476

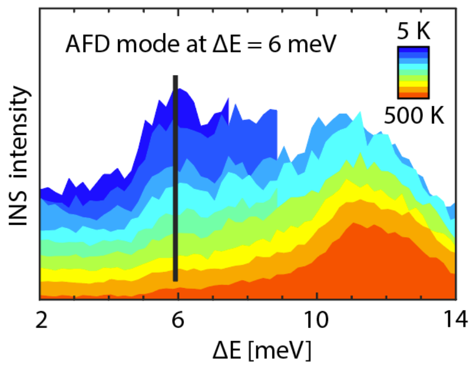
Unraveling the ground-state structure of $BaZrO_3$ by neutron scattering experiments and first-principles calculations

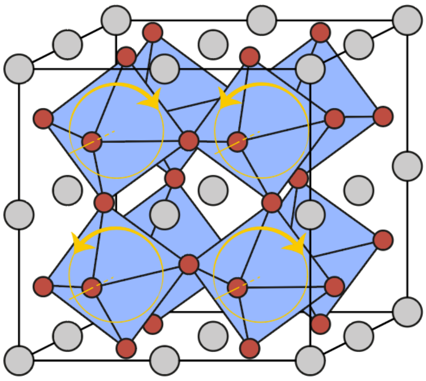
A. Perrichon[a,b], E.J. Granhed[b], G. Romanelli[a], A. Piovano[c], A. Lindman[b], P. Hyldgaard[b], G.Wahnström[b], M. Karlsson[b]
[a] ISIS Neutron and Muon Source, RAL, STFC, Harwell, United Kingdom,
[b] Chalmers University of Technology, Göteborg, Sweden,
[c] Institut Laue-Langevin, Grenoble, France

adrien.perrichon@stfc.ac.uk

The all-inorganic perovskite barium zirconate $BaZrO_3$ is widely used in the field of energy materials, with numerous proton-conducting oxide materials being obtained by, for instance, acceptor-doping of the Zr-sites [1]. However, fundamental questions remain surrounding the crystal structure of $BaZrO_3$, especially regarding its ground-state structure.

While diffraction techniques indicate a cubic structure all the way down to T = 2 K [2], several first-principles phonon calculation studies based on density functional theory indicate an imaginary (unstable) phonon mode due to the appearance of an antiferrodistortive (AFD) transition associated with rigid rotations of ZrO_6 octahedra. The first-principles calculations are highly sensitive to the choice of exchange-correlation functional and, using six well-established functional approximations, we show that a correct description about the ground-state structure of $BaZrO_3$ requires the use of hybrid functionals. The ground-state structure of $BaZrO_3$ is found to be cubic, which is corroborated by experimental results obtained from neutron powder diffraction, inelastic neutron scattering, and neutron Compton scattering experiments [3].





References

[1] K.D. Kreuer, Proton-Conducting Oxides, Annu. Rev. Mater. Res. **33**, 333 (2003)
[2] A.R. Akbarzadeh et al., Combined Theoretical and Experimental Study of the Low-Temperature Properties of $BaZrO_3$, Phys. Rev. B **72**, 205104 (2005)
[3] A. Perrichon et al., Unraveling the Ground-State Structure of $BaZrO_3$ by Neutron Scattering Experiments and First-Principles Calculations, Chem. Mater. **32**, 2824 (2020)

From PEF to PBF: a computational spectroscopy study of furandicarboxylate-based polyesters

Paulo Ribeiro-Claro[a]

[a]*Department of Chemistry and CICECO, University of Aveiro, Aveiro, Portugal*

prc@ua.pt

Global awareness of the finite nature of fossil resources rose the interest on bio-based polyesters and, in particular, those derived from 2,5-furandicarboxylic acid (FDCA). This work describes a computational spectroscopy study of the furandicarboxylate-based polyesters poly(ethylene 2,5-furandicarboxylate) (2,5-PEF)[1], poly(ethylene 2,4-furandicarboxylate) (2,4-PEF) [2], and poly(butylene 2,5-furandicarboxylate) (2,5-PBF) [3]. A thorough computational spectroscopic study – including infrared, Raman and inelastic neutron scattering spectroscopy, combined with discrete and periodic density functional theory calculations – allowed the identification of dominant structural motifs in the amorphous and crystalline regions on polymer samples. For 2,5-PEF [1], the polymeric chains adopt, in the crystalline domains, an energetically unfavorable extended all-trans geometry , which is stabilized by a network of C-H...O bonds linking adjacent chains. This stabilization is not possible for 2,4-PEF [2], due to a random distribution of monomer orientation along the polymer chain. In the absence of C−H···O stabilization, the ordered crystalline arrangement is disfavored and polymer chains prefer to solidify into an amorphous mess. In what concerns 2,5-PBF [3], discrete calculations and vibrational spectroscopy of semi-crystalline and amorphous samples strongly support the predominance of *gauche,trans,gauche* conformations of the butylene glycol fragment in both the crystalline and amorphous domains. The amorphous domains are dominated by *syn,syn* conformations of the furandicarboxylate fragment, while in the crystalline domains the *anti,anti* forms prevail. A possible crystalline structure – built from these conformational preferences and including a network of C-H...O hydrogen bond contacts – was optimized using periodic density functional theory.

References

[1] Inside PEF: Chain Conformation and Dynamics in Crystalline and Amorphous Domains, C. F. Araujo, M.M. Nolasco, Paulo J. A. Ribeiro-Claro, S. Rudic, A.J.D. Silvestre, P. D. Vaz, and A. F. Sousa. Macromolecules, 51 (2018) 3515–3526 (DOI: 10.1021/acs.macromol.8b00192).

[2] Asymmetric monomer, amorphous polymer? Structure-property relationships in 2,4-FDCA and 2,4-PEF, M.M. Nolasco, Catarina F. Araujo, S. Thiyagarajan, S. Rudic, P. D. Vaz, A. J. D. Silvestre, P. J. A. Ribeiro-Claro, A. F. Sousa. Macromolecules, 53 (2020) 1380-1387 (DOI: 10.1021/acs.macromol.9b02449)

[3] From PEF to PBF: what difference does the longer alkyl chain make? M.M. Nolasco, Paulo Ribeiro-Claro, Pedro D. Vaz, Catarina F. Araújo, S. Rudic, A. F. Sousa1, A. Silvestre1, L. Rodrigues (in press).

Abstract: 028

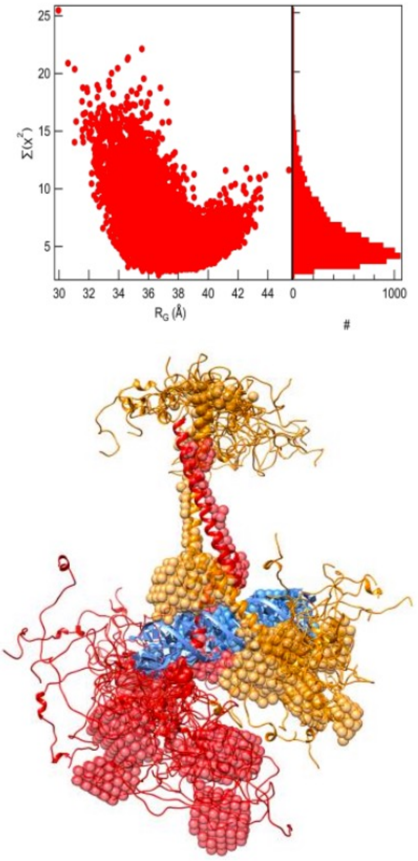
Small-angle-scattering and biophysical analysis of an extended MYC- MAX-DNA complex including the conserved MYC-MBIV region

Francesca Caporaletti[a,b], Vamsi Krishna Moparthi[b], Dean Derbyshire[a], Anne Martel[b], Maria Sunnerhagen[a]

[a]Linkoping University, Linkoping, Sweden,
[b]Institute laue Langevin, Grenoble, France

Caporaletti.franc@gmail.com

The Myc family oncogene are deregulated in more than 50% of hu- man cancers [1]. Myc is a central hub in almost every oncogenic aspect, regulating apoptosis proliferation, differentiation and metabolism [2,3]. Incapacitating MYC directly holds promise to be a powerful and effective method to treat several cancer types. One alternative would be the interruption of the Myc-Max complex because Myc strictly depends on its partner Max to regulate gene transcription and thereby convey a range of cellular signalling pathways [4]. However, hitherto, strategies to interrupt a well-conserved DNA-binding MYC-MAX complex comprising a small c-terminal region of MYC have not led to progress in the clinic [1]. In this work we describe the structural and biophysical properties of a larger MYC fragment in complex with MAX and DNA, using a combination of small-angle X-ray and neutron scattering together with the biophysical characterization of structure, stability and DNA binding. We show that including the MBIV region of MYC significantly stabilizes the MYC-MAX-DNA complex and leads to increased secondary structure content, but that the localization of the MYC-MBIV region is still predominantly disordered, both intrinsically and with respect to the core DNA-anchored MYC-MAX heterodimer fold. Characterization of this extended MYC-MAX-DNA complex contributes an extended platform for therapeutic targeting of the MYC oncoprotein.



Results of the analysis performed with the 6 different scattering conditions and 10000 all-atoms models generated with Rosetta. Superimposition between the ab-initio monsa and the best 1% of the all-atoms models over the 6 scattering profiles.

References

[1] Albihn A et al. MYC in oncogenesis and as a target for cancer therapies. Advances in cancer research. 2010 Jan 1;107:163-224.

[2] Nie Z et al. c- Myc is a universal amplifier of expressed genes in lymphocytes and embryonic stem cells. Cell. 2012 Sep 28;151(1):68-79.

[3] Meyer N et al. Reflecting on 25 years with MYC. Nature Reviews Cancer. 2008 Dec;8(12):976-90. [4] Chen H, Liu H, Qing G. Targeting oncogenic Myc as a strategy for cancer treatment. Signal transduction and targeted therapy. 2018 Feb 23;3(1):1-7.

Abstract: 030

Structural changes during annealing of core-shell latex colloidal thin films

Stephan V. Roth[a,b], *Calvin J. Brett*[a,b], *Alexandros E. Alexakis*[b], *Joakim Engström*[b], *Alexei Vorobiev*[c,d], *Eva Malmström*[b]

[a]*Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany*
[b]*KTH Royal Institute of Technology KTH, Stockholm, Sweden*
[c]*Department for Physics and Astronomy, Uppsala University, Uppsala, Sweden*
[d]*Institute Laue-Langevin, Grenoble, France*

stephan.roth@desy.de

The use of latex nanoparticles (NPs) in colloidal coatings is very interesting due to their easy water-processability and wide industrial application range, already in dispersive paints and adhesives [1]. Latexes are dispersed stable colloidal polymeric particles in water phase, commonly synthesized by emulsion polymerization. Block copolymer latexes fabricated via polymerization-induced self-assembly (PISA) form core-shell particles with tailored corona properties to interact with specific surfaces such as cellulose or silica. Since the properties of colloidal coatings are given by the nanostructure and morphology of the film and its surface [2], understanding the film formation, self-assembly and temperature stability of latex coating systems is of great interest.

We used neutron reflectometry (NR) and grazing incidence small-angle neutron scattering (GISANS) at SUPERADAM to follow the density gradient and surface morphology of latex films with increasing annealing temperature (room temperature to 140°C). We used spray deposited thin films [2] and latex NPs consisting of a poly(methyl methacrylate) (PMMA) core and a 2-dimethylaminoethyl methacrylate (PDMAEMA) shell polymer. We synthetized two distinct sizes (45 nm, 110 nm) of NPs and prepared the samples either with protonated or deuterated PMMA core. The substrate was silicon, and we deposited different NPsub-monolayers or multilayer structures. For the multilayer films we deposited a thin film of nanocellulose [3] as porous substrate material and then studied how the nanoparticles penetrate in this porous media and how the temperature affects the NPs within. Especially the NR data directly show the stratification of the colloidal film at elevated temperatures which is related to the collapse of the core and the shell. This is directly correlated to the morphological changes of the core and shell observed by GISANS.

References

[1] Leng et al., J. Mater. Chem. C, 2018, 6, 2396
[2] Engström et al., Adv. Funct. Mater., 2020, 30, 1907720
[3] Brett et al., Macromolecules, 2019, 52, 4721

Abstract: 032

Assessing the structural dynamics of chloromethane derivatives through INS

Mariela Nolasco[a], Pedro Vaz[a], Paulo Ribeiro-Claro[a], Stewart Parker[b]

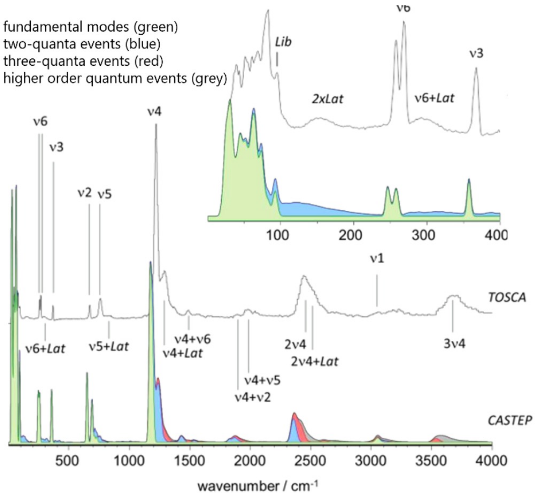
[a]CICECO- Instituto de Materiais de Aveiro, Departamento de Química, Universidade de Aveiro,
P- 3810-193 Aveiro, Portugal
[b]ISIS Neutron & Muon Source, STFC Rutherford Appleton Laboratory, Didcot, Oxfordshire OX11 0QX, UK

mnolasco@ua.pt

In this work, following our continuing efforts, the structural dynamics of chloromethane derivatives is evaluated through our computational spectroscopy approach by comparing experimental INS spectra with the corresponding simulated spectra obtained from periodic DFT calculations (performed for different systems such as cyclopentanone and benzaldehyde derivatives [1,2].

To the best of our knowledge, there are no reports on the Inelastic Neutron Scattering (INS) spectra of these compounds. INS spectroscopy provides a unique assessment of the structural dynamics that is not amenable from its optical counterparts, infrared and Raman spectroscopies. Large amplitude/low wavenumber modes, including lattice modes, usually problematic for optical spectroscopy, tend to yield intense bands in INS spectra. The excellent agreement between experimental and calculated spectra (exemplified here for CHCl3) allows a confident assignment of the vibrational features, including not only the molecular fundamental modes, but also lattice and combination modes.

For the fundamental modes, calculations correctly predict the split of the ν6 mode (observed at *ca.* 257- 268 cm^{-1}) and the low intensity of the ν1 (observed at *ca.* 3060 cm^{-1}). More impressive is the remarkable prediction of the multi-quanta sequence based on ν4, spanning up to 3680 cm^{-1}. Besides the 1x ν4, 2x ν4 and 3x ν4 sequence, there are also two-quanta combinations of ν4 with ν2, ν5 and ν6. Both translational and librational modes appear with significant contribution to multi-quanta modes. This effect may be related with the stronger C- H···Cl contacts in CHCl3.



References

[1] Vaz, P.D.; Nolasco, M.M.; Ribeiroi-Claro, P.J.A. Chemical Physics 427 (2013): 117

[2] Nolasco, M.M.; Ribiero-Claro, P.J.A.; Vaz, P.D. Materials 15 (2022): 4745

Methanol diffusion in H-ZSM-5 catalysts as a function of loading and Si/Al ratio: A classical molecular dynamics study

<u>Claire-Louise M. Woodward</u> [1], *Alexander J. Porter* [1], *Katie S.C. Morton* [1,2], *Alexander J. O'Malley* [1,2,]

[1]University of Bath, Bath, UK,
[2]ISIS Neutron and Muon Source, Oxford, UK

clw89@bath.ac.uk

The monetisation of natural gas and the high demand for light olefins has resulted in the zeolite catalysed methanol-to-hydrocarbons (MTH) process becoming of great industrial and academic interest. The mobility of methanol molecules to and from the active site can be affected by factors including Si/Al ratio (and thus acid site density) and methanol loading, having a significant impact on catalyst activity/selectivity. Classical molecular dynamics is a powerful theoretical tool to probe the nanoscale mobility of methanol in zeolite catalysts, especially in combination with quasielastic neutron scattering (QENS), where QENS observables may be directly reproduced from the atomic trajectories, or more broadly, diffusion coefficients and qualitative behaviours may be obtained/observed to aid in the data analysis. While the effect of acid site presence, and the comparison of two extreme Si/Al ratios has been studied either experimentally [1] or with computational methods [2], parametric studies of nanoscale methanol diffusion across a range of Si/Al ratios are yet to take place – particularly with different methanol loadings.

Here, methanol diffusion in the Brønsted acidic industrial catalyst H-ZSM-5 has been modelled using classical MD at 373 K, 423 K and 473 K, using loadings of 3 and 5 molecules of methanol per unit cell (MPUC), in frameworks with Si/Al = 15, 47, 95, 191 [3]. While the lower loading exhibits higher diffusivity, self-diffusivities increase at both loadings between Si/Al = 15 and 95, after which they are independent of composition (only deviating by ~1.5×10^{-10} m^2s^{-1} over the remaining range). This deviation leads to an 'r-shaped' curve of diffusivity as a function of Si/Al ratio. This suggests that if less than 1 BAS per unit cell (Si/Al > 95) is present, no further trend is observed in methanol diffusivity with Si/Al ratio, which may be attributed to the infrequency of methanol-BAS H-bonding interactions, and the steric hindrance of the pore structure becomes the most significant factor in overall methanol mobility.The trend in diffusivity with Si/Al ratio is explained in terms of methanol-acid site interactions, while the trend with loading is explained in terms of methanol-methanol interactions and the resulting methanol structure in the catalyst pores. QENS observables were reproduced from these classical MD simulations (the intermediate scattering function, the EISF and QENS broadenings) and will be compared directly with future QENS experiments to ensure the validity of the employed MD model. These will lay the foundation for future studies into the effect of composition of active species behaviour in future MTH catalysts.

References

[1] T. Omojola, I.P. Silverwood, A.J. O'Malley, *Catal. Sci. Technol.*, **10**, 4305-4320, (2020)

[2] C.H. Botchway, R. Tia, E. Adej, A.J. O'Malley, N.Y. Dzade, C. Hernandez-Tamargo, N.H. de Leeuw, *Catalysts*, **10**, 1-18, (2020)

[3] C.-L.M. Woodward, A.J. Porter, K.S.C. Morton, A.J. O'Malley, *Catal. Commun.*, **164**, 106415, (2022)

Local and nanoscale water behaviour in zeolites

A.J. Porter[1]*, A.J. O'Malley*[1]

[1]Centre for Sustainable and Circular Technologies, Department of Chemistry, University of Bath, BA2 7AY

AP2110@bath.ac.uk

Water behaviour within zeolites is a highly important field owing to their wide application in water purification, molecular separation, and catalysis. Across these processes, water behaviour can impact the catalytic and adsorption/separation properties and thus experimental techniques such as quasielastic neutron scattering (QENS) as well as computational techniques such as molecular dynamics (MD), which probe similar time and length scales, are pivotal to probe water dynamics. A detailed understanding of such dynamics as a function of zeolite structure and composition will allow the design and optimisation of zeolite materials. Specifically, the effect of the Si/Al ratio – which controls acid site density within the zeolite framework, is of great interest.

The behaviour of water has been probed in acidic H-FAU [1] and H-MFI type zeolites as a function of Si/Al ratio (5 to siliceous and 15 to siliceous respectively) employing QENS and MD. Water diffusivity, on the order of $1 – 10 \times 10^{-10}$ m^2s^1, was found to decrease with Si/Al ratio in both systems– figure 1. This is primarily driven by strong interactions between water molecules and the Brønsted acid sites – figure 2 – with residence times of up to 5 ps. Water loading, in FAU, was also found to have a significant effect where diffusivity increased significantly by a factor of ~7 towards a maximum diffusivity. QENS data from the MFI systems were best fit to the behaviour of jump diffusion confined to a sphere (of ~4 Å such as a channel intersection) with jump distances around 3 Å. Similar behaviour was observed in both the Si/Al = 15 and 40 systems whilst the Si/Al = 140 system showed markedly different behaviour with much larger confined radii and significantly shorter residence times – suggesting more long range translational diffusion. Diffusivity of water in the MFI systems was similar to that of its FAU counterparts at comparable loadings via MD analysis. Qualitative and quantitative agreement was seen between the QENS and MD data for water in H-MFI.

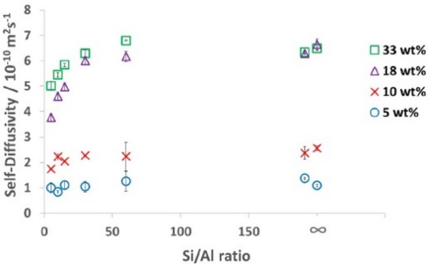


Figure 1. Diffusion coefficients of water at 5, 10, 18, and 33 wt % loading in the fully siliceous and Si/Al = 191, 60, 30, 15, 10, and 5 zeolite HY systems

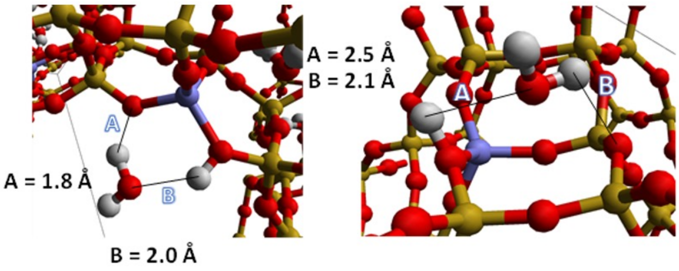


Figure 2. Two modes of water interacting with a Brønsted site in zeolite HY.

References

[1] A. J. Porter and A. J. O'Malley, *The Journal of Physical Chemistry C*, **125** (21), 11567-11579(2021).)

Abstract: 037

Protein diffusion during non-classical crystallization processes

Christian Beck[a,b], *Ralph Maier*[b], *Fajun Zhang*[b], *Felix Roosen-Runge*[c], *Frank Schreiber*[b], *Tilo Seydel*[a]

[a]*Institut Max von Laue-Paul Langevin, Grenoble, France*
[b]*University Tübingen, Tübingen, Germany*
[c]*University Malmö, Malmö, Sweden*

christian.beck@uni-tuebingen.de

High quality protein crystals are important for structure determination of proteins and for purification processes. However, different crystallization processes are known and a systematic, complete understanding of underlying process is still missing. In addition to structural characterization and the crystallization kinetics, the investigation of the diffusive processes is necessary to comprehend the dynamics of crystal formation.

High resolution neutron spectroscopy offers access to the short-time diffusive properties of protein solutions [1]. Nowadays, thanks to instrumental developments, kinetically changing samples can be followed with time dependent measurements. By applying time-dependent neuton backscattering and neutron spin echo, it is possible to follow the short-time collective and self diffusion of different components of the solution during the crystallization process (Figure 1) [2].

Here, we present a study on the diffusive processes during the nonclassical crystallization process using dissolved beta-lactoglobulin (BLG) in the presence of $CdCl_2$ at different concentrations. The results will be compared with complementary small angle scattering data (Figure 2) [3] as well as classical crystallization processes investigated earlier for a different system [2].

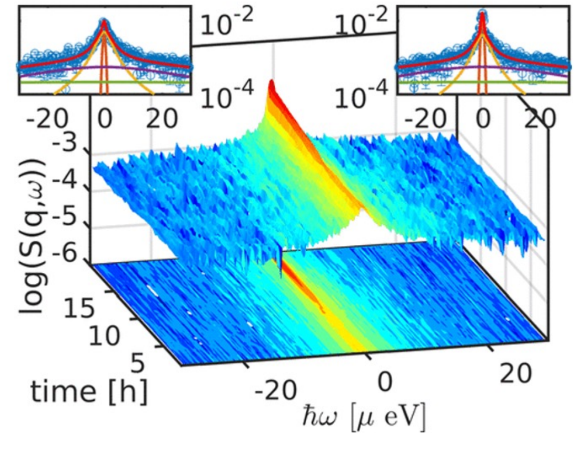


Figure 1: Time-dependent QENS measurements of a classical crystallization process [2].

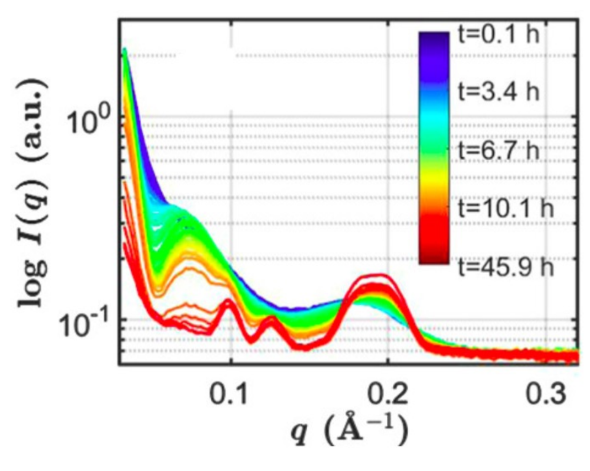


Figure 2: Real time SANS data of a nonclassical crystallization process [3].

References

[1] M. Grimaldo et al., Quart. Rev. Biophys. **52** (2019) e7, 1
[2] C. Beck et al., Cryst. Growth Des. **19** (2019) 7036
[3] R. Maier et al., Cryst. Growth Des. **21** (2021) 6971

Abstract: 038

NUrF: streamlining liquid handling & data processing and analysis for SANS beamlines

Gudrun Lotze[a], *Cedric Dicko*[b], *Judith Houston*[c], *Simon Heybrock*[c], *Adrian Rennie*[a]

[a]*Uppsala University, Chemistry, Uppsala, Sweden*
[b]*Lund University, Chemistry, Lund, Sweden*
[c]*European Spallation Source ESS, Lund, Sweden*

gudrun.lotze@kemi.uu.se, cedric.dicko@tbiokem.lth.se

Automated or robotic liquid handling systems are devices that perform various liquid handling tasks in laboratories, including pipetting, sample preparation, and cuvette washing. Those devices are now standard at most small-angle X-ray scattering beamlines [1]. They help to save on labour and free up resources to focus on analysis instead of repetitive liquid handling tasks.

However, the application of automated or robotic liquid handling systems at small- angle neutron scattering beamlines is in its infancy. The most recent and successful system involves (i) an in-line liquid chromatography system to separate and subsequently measure biomacromolecules and their complexes [2]; and (ii) microfluidic cells to perform reactions [3]. A significant difficulty is the optimization of sample volume, concentration and deuteration state.

We recently developed combined small-angle neutron scattering with UV-visible and fluorescence spectroscopies, with the addition of in-line density measurements: the NUrF [4].

On the software side, we propose a new outline for hdf5 files for neutron sources using the NUrF experimental configuration. The file format follows the NeXus data standards ensuring that data can be loaded without any specific file loader software now and in 20 years' time [5]. The new hdf5 file structure integrates smoothly with the scipp Python library [6] facilitating data processing and future analysis.

Overall, this contribution illustrates how automatic liquid handling needs to go hand in hand with automatic data processing, visualization, and analysis to make efficient use of neutron beamtime and to reveal unexpected events in the sample.

References

[1] Round A, Felisaz F, Fodinger L, Gobbo A, Huet J, Villard C, et al., Acta Crystallographica Section D - Structural Biology. 2015; 71(1), 67–75, doi:10.1107/S1399004714026959

[2] Jordan A, Jacques M, Merrick C, Devos J, Forsyth VT, Porcar L, et al., J. Appl. Cryst. 2016; 49(6), 2015-2020, doi:10.1107/S1600576716016514.

[3] Adamo M, Poulos AS, Miller RM, Lopez CG, Martel A, Porcar L, et al., Lab on a Chip 2017; 17(9): 1559-1569, doi: 10.1039/C7LC00179G.

[4] Dicko C, Engberg A, Houston JE, Jackson AJ, Pettersson A, Dalgliesh RM, et al., Review of Scientific Instruments. 2020; 91(7), 075111, doi: 10.1063/5.0011325.

[5] https://scipp.github.io/scippnexus/

[6] Heybrock S, Arnold O, Gudich I, Nixon D, Vaytet, N, Journal of Neutron Research, 2020; 22(2-3): 169-181, doi: 10.3233/JNR-190131, https://scipp.github.io/

Abstract: 039

Probing model lignin monomers within zeolite catalysts of varying compositions

K. S. C. Morton[a,b], *A. O'Malley*[a], *J. Armstrong*[c]

[a]*University of Bath, Bath, UK*
[b]*ISIS Neutron and Muon Source, Oxford, UK*

katie.morton@stfc.ac.uk

Lignin is an abundant component of lignocellulosic biomass that has significant potential as a renewable feedstock. Zeolite catalysts will be of great importance for lignin upgrading, with mixed biomass and crude oil feeds having already been commercially employed [1]. Probing the behaviour of model lignin monomers, such as the cresol isomers, within microporous catalyst frameworks is crucial for designing and optimizing the reaction for products such as gasoline other precursors for drugs, dyes and polymers [2].

Initially, QENS was applied to investigate bulk liquid cresol diffusion from 340-390 K to aid the parameterization of molecular dynamics (MD) simulations. The linear *para*-isomer exhibited faster rates of both isotropic rotation and translational jump diffusion, in comparison to *meta*-cresol. Modelled translational self-diffusion coefficients indicated the same trend.

Upon dosing the cresols into zeolites, models of isotropic rotation fit closely to the experimental elastic incoherent scattering function (EISF), shown in Figure 1. A greater population of mobile cresol was observed in the larger pores of zeolite HY (7.4 Å in diameter) compared to the pores of HBEA (6.7 Å in diameter).

Inelastic neutron scattering (INS) was used to probe the zeolite acid site to cresol interaction strengths by analysing changes in the vibrational frequencies of the cresol molecules. The diminishing of peaks relating to the hydroxyl bending of the cresol (~350 cm^{-1}) when inside the zeolite indicates its adsorption to acid sites, which is also essential for catalysis.

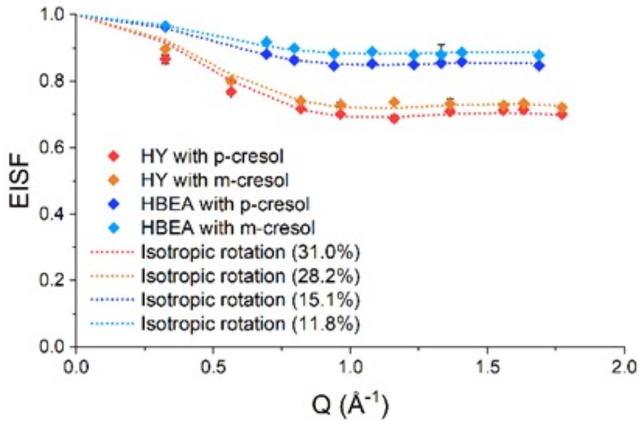


Figure 1. Isotropic rotation models fit to the EISF of m-cresol and p-cresol in HY and HBEA at 370 K.

References

[1] P. de Rezende, A. de Almeida, M. B. B. Marlon, F. L. Mendes, L. C. Casavechia, M. S. Talmadge, C. M. Kinchin and H. L. Chum, Fuel, **188**, 462-473, (2017).

[2] A. T. To and D. E. Resasco, Appl. Catal. A-Gen., **487**, 62–71, (2014).

Structural study of mouse translocator protein (mTSPO) in different amphiphilic environments.

C. Saade[a], S. Finet[b], F. Bonneté[c], J-J Lacapère[d], and S. Combet[a]

[a]*Laboratoire Léon-Brillouin (LLB), UMR 12 CEA-CNRS, CEA Saclay.*
[b]*Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie (IMPMC), UMR 7590, Sorbonne University.*
[c]*Institut de Biologie Physico-Chimique (IBPC), UMR 7099 CNRS, Universite´de Paris.*
[d]*Laboratoire des BioMolécules (LBM), UMR 7203 CNRS, Sorbonne University.*

Christelle.saade@cea.fr

The translocator protein TSPO is a functionally important transmembrane protein, mainly localized in the outer mitochondria membrane, with a strong pharmacological interest. For mammalian TSPO, no crystals have yet been obtained and a high-resolution structure remains challenging especially in absence of ligands[1].

We study the effects of different amphiphilic environments on mouse TSPO (mTSPO) structure, using a new strategy combining small-angle X ray and neutron scattering approaches (SAXS/SANS) with ab initio modelling. Our objective is to determine the structure/function of mTSPO in: (i) SDS, its extraction and purification detergent ; (ii) in DPC, the detergent used to solve its structure by liquid NMR, but in presence of a stabilizing ligand (2MGY:PDB) [2] that stiffens the protein; (iii) and in DPC/DMPC mixtures, to mimic a more biomimetic lipid environment [3]. Such a structural information is crucial to determine the optimal conditions where mTSPO is stable and functional for further high-resolution studies (X-ray crystallography, NMR, cryo-EM), which are necessary to develop new molecules for imaging, diagnostics, and therapeutics.

Using SANS and the contrast-matching technique, which makes the detergent stealth to probe only the protein, we have recently shown that mTSPO conformation is partially unfolded and flexible in SDS. In contrast, in DPC the protein is much more compact and structured but still significantly different from the ligand-binding NMR structure (Figure) [4].

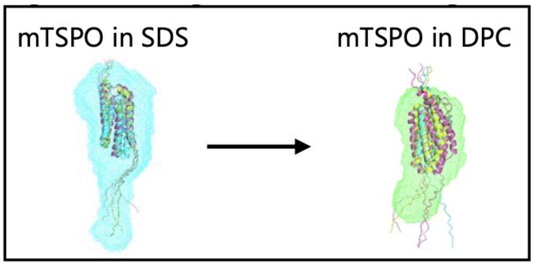


Figure. Solution structures of mTSPO without ligand in SDS and DPC, compared to its ligand-binding NMR structure (2MGY:PDB).

References

[1] J. J. Lacapere, L. Duma, S. Finet, M. Kassiou, and V. Papadopoulos, "Insight into the Structural Features of TSPO: Implications for Drug Development," Trends Pharmacol.
[2] Ł. Jaremko, M. Jaremko, K. Giller, S. Becker, and M. Zweckstetter, "Structure of the mitochondrial translocator protein in complex with a diagnostic ligand," Science.
[3] J. J. Lacapère et al., "Structural and functional study of reconstituted peripheral benzodiazepine receptor," Biochem. Biophys. Res. Commun.
[4] S. Combet, F. Bonnete, S. Finet, A. Pozza, C. Saade, A. MartelL, A. KoutsiouBbas, and J.J Lacapere. "Effect of amphiphilic environment on the solution structure of mouse TSPO translocator protein", Biochemistry (submitted)

Structural disorder and magnetic correlations in non-Kramers $Tb_2Hf_2O_7$ pyrochlore

V. Porée[a], E. Lhotel[b], P. M. Derlet[a], T. Fennell[a], M. Kenzelmann[a] & R. Sibille[a]

[a]Paul Scherrer Institut, Switzerland
[b]Institut Néel, CNRS, France

victor.poree@psi.ch

The identification of possible mechanisms to stabilise Quantum Spin Liquid (QSL) phases [1] is of prime interest. In the case of non-Kramers pyrochlores, the introduction of transverse fields in the Hamiltonian was proposed theoretically [2-3], and argued experimentally [4], as a possible route to promote quantum fluctuations in spin ices [2-3].

Such physics was also proposed to be at work in $Tb_2Hf_2O_7$ [5], where experimental investigations have revealed the stabilisation of a spin liquid state despite a massive amount of disorder in the crystal, with about half of the terbium sites characterised by a defective coordination sphere. Here we present a detailed structural analysis of this material based on neutron pair distribution function (PDF) data, which we use to quantitatively relate to the magnetic properties.

We show how the big-box modelling of such a disordered material can be used to understand its crystal-electric field spectrum. The distribution of single-ion states, extracted from our model box including 2000 terbium sites, provides additional insights to explain the peculiar correlated magnetic ground state observed in $Tb_2Hf_2O_7$. We link these results with specific heat, ac-susceptibility, muon spin-relaxation and low-energy neutron spectroscopy data, allowing to further understand the partial spin freezing of about 15% of the sites occurring deep in the correlated phase. Our analysis indicates that this proportion of frozen degrees of freedom matches the number of doublets that are split by an energy scale exceeding that of the dominant interactions. More generally, we thus further demonstrate that for most of the spin system, where the doublet splitting is substantial but weaker than dominant interactions, pseudo-doublets are correlated to form a spin liquid.

References

[1] L. Balents, Nature 464 199 (2010)
[2] L. Savary and L. Balents, Phys. Rev. Lett. 118, 087203 (2017)
[3] O. Benton, Phys. Rev. Lett. 121, 037203 (2018)
[4] J.-J. Wen et al., Phys. Rev. Lett. 118, 107206 (2017)
[5] R. Sibille et al., Nat. Commun. 8, 892 (2017)

Abstract: 044

Structural and dynamical properties of liquid chromophores

Eric Lindgren[a], *Paul Erhart*[a]

[a]*Department of Physics, Chalmers University of Technology, Gothenburg, Sweden*

ericlin@chalmers.se

Liquid chromophores such as perylene and its derivatives constitute an important class of materials, with applications ranging as solvent-free dyes to increasing the efficiency of solar cells via photon conversion. Their structural and in particular dynamical behaviour on the molecular level is not very well known, yet crucial for their optical properties. In this work, we use molecular dynamics (MD) simulations to investigate the structural and dynamical properties of perylene and two of its derivatives (perylene-ethyl and perylene-diimide). Specifically, we extract the static and dynamical structure factors and the current correlation functions for various temperatures as well as for two different initial structural models of the systems, which in principle allows us to establish a direct link to experimental studies. Motivated by recent experimental work [1], we consider two structural models that differ with respect to the relative orientation of the molecules. Whereas in model 1 the molecules are more or less randomly oriented, model 2 features domains with pronounced pi-pi stacking. The latter configuration corresponds to a simplified representation of the supramolecular aggregates that some extended perylene derivatives have been observed to form experimentally [2,3,4]. The ability of perylene systems to assume either or both of these structures depends on the sidegroups and can also be affected by mixing different derivatives. Using the two molecular variants indicated above, we have now established our simulation protocol and verified the basic premise of this project. For the molecules considered so far we only find the disordered state (model 1). Next we will extend our study to derivatives with larger sidegroups and mixtures thereof.

References

[1] Sandra Hultmark et al. “Vitrification of Octonary Perylene Mixtures with Ultralow Fragility”. In: Science Advances 7.29 (July 2021), eabi4659. ISSN: 2375-2548. DOI: 10.1126/sciadv.abi4659

[2] Max Burian et al. “Structural and Optical Properties of a Perylene Bisimide in Aqueous Media”. In: Chemical Physics Letters. Ahmed Zewail (1946-2016) Commemoration Issue of Chemical Physics Letters 683 (Sept.2017), pp. 454–458. ISSN: 0009-2614. DOI: 10.1016/j.cplett.2017.03.056.

[3] Ari Laiho et al. “Macroscopically Aligned Ionic Self-Assembled Perylene-Surfactant Complexes within a Polymer Matrix”. In: Advanced Functional Materials 18.13 (2008), pp. 1890–1897. ISSN:1616-3028. DOI: 10.1002/adfm.200701496.

[4] Yongwei Huang et al. “Helical Supramolecular Aggregates, Mesoscopic Organisation and Nanofibers of a Perylenebisimide–Chiral Surfactant Complex via Ionic Self-Assembly”. In: Journal of Materials Chemistry19.16 (Apr. 2009), pp. 2356–2362. ISSN: 1364-5501. DOI: 10.1039/B817838K.

Probing topological interactions in polymers under shear

Manuchar Gvaramia[a], Philipp Gutfreund[b], Lisa Höglund[c,], Ayan Samanta[c], Max Wolff[a]

[a]Department for Physics and Astronomy, Uppsala University, Regementsvägen 1, SE-75120 Uppsala, Sweden
[b] Institut Laue-Langevin,71 Avenue des Martvrs, 38000 Grenoble, France
[c]Department for Chemistry, Macromolecular chemistry, Uppsala University, Regementsvägen 1, SE-75120 Uppsala, Sweden

manu.gvaramia@gmail.com

Wormlike micelles may serve as a model system for linear polymers and are studied extensively. Micelles, unlike polymers, can break and reform. Their self-assembled structure makes them unique in applications where high shear rates may be encountered, such as drag reduction, and as a templates for materials synthesis. The rheological properties of viscoelastic materials can be described by the Maxwellian model.

In this presentation we will present the influence of CTAB-NaSal solutions composition on the rheology of wormlike micelle and pave the road for microscopic investigation by neutron scattering. Fig. 1 shows the linear viscoelastic rheology of cationic wormlike micellar solution which has been shown to be Maxwellian, exhibiting a single dominant relaxation time.

To study the microscopic dynamics of wormlike micelles, they can be measured by neutron spin echo spectroscopy (NSE). However, such measurements under shear are challenging as Doppler scattering may depolarize the beam. In this presentation we will present a new dedicated sample cell to allow Rheo-NSE studies. We show resolution measurements and compare them to theoretical predictions of Doppler scattering. We show that our shear cell allows to address Fourier times up to 140ns at Q values of 0.157 Å^{-1} and shear rates of up to 166 s^{-1}.

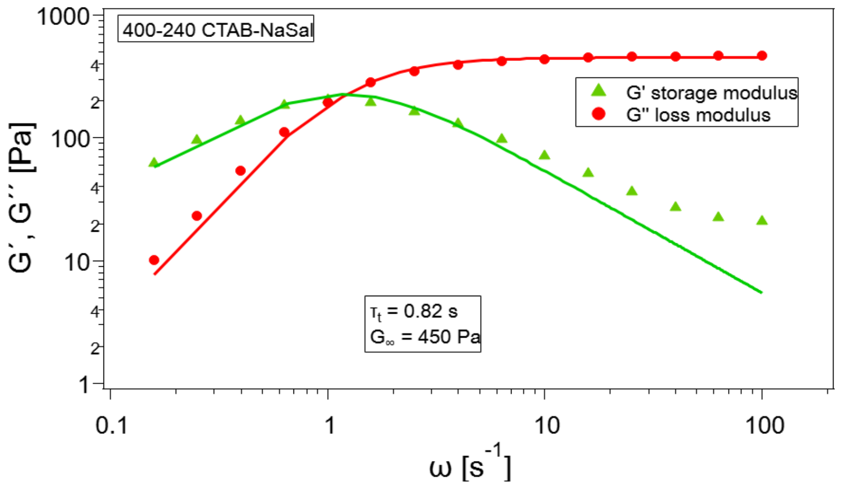


Fig 1. Storage and loss modulus of CTAB-NaSal based wormlike micelles fitted with a Maxwellian model.

Bayesian refinement of diffraction data

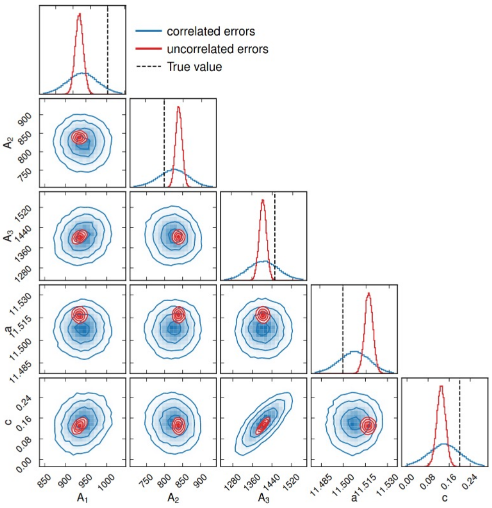
Erik Fransson[a], Piotr Rozyczko[b], Andrew Sazonov[b], Thomas Holm Rod[b], Paul Erhart[a]

[a]Chalmers University of Technology, Gothenburg, Sweden,
[b]European Spallation Source ERIC, PO BOX 176, SE-221 00 Lund, Sweden

erikfr@chalmers.se

In this work we present a Bayesian methodology to analyze diffraction patterns. The Bayesian framework allows one to define a probability distribution over, e.g., crystallographic parameters which can be sampled using Monte Carlo (MC) simulations, as demonstrated in [1]. This can yield more information about the model parameters than regular least-square minimization (as in Rietveld refinement) in terms of uncertainties and correlations. Additionally, it allows for encoding any prior knowledge about the sample in a rigorous statistical framework, and allows for modeling of any error correlation in the likelihood.

We employ the open-source software EasyDiffraction [2] to simulate diffraction patterns, and use the package emcee to run MC. This approach is benchmark on the neutron powder diffraction data for $PbSO_4$ as well as for some synthetic data. Specifically we find that taking into account correlations in errors can be crucial in order to find correct distributions (i.e., uncertainty) of parameters.



We aim to provide this type of analysis as a small collection of jupyter notebooks in order for allow users to apply this methodology to their application. Hopefully this can lead to a better understanding of the distribution over crystallographic parameters and how the basic assumptions regarding errors affect these distributions.

References

[1] Fancher, C. M., Han, Z., Levin, I., Page, K., Reich, B. J., Smith, R. C., Wilson, A. G., & Jones, J. L. (2016). Use of Bayesian Inference in Crystallographic Structure Refinement via Full Diffraction Profile Analysis. In Scientific Reports (Vol. 6, Issue 1)

[2] https://easydiffraction.org/

Abstract: 051

Signatures for Berezinskii-Kosterlitz-Thouless critical behavior in the planar antiferromagnet $BaNi_2V_2O_8$

Ekaterina Klyushina[a,b], *J. Reuther*[c,b], *L. Weber*[d], *A. T. M. N. Islam*[b], *J. S. Lord*[e], *B. Klemke*[b], *M. Månsson*[f,g], *S. Wessel*[d] *and B. Lake*[b]

[a]*Lund University, Lund, Sweden*
[b]*Helmholtz-Zentrum Berlin für Materialien und Energie, Berlin, Germany*
[c]*Freie Universität Berlin, Berlin, Germany*
[d]*RWTH Aachen University, Aachen, Germany*
[e]*ISIS facility, STFC Rutherford Appleton Laboratory, Didcot, United Kingdom*
[f]*KTH Royal Institute of Technology, Stockholm, Sweden*
[g]*Paul Scherrer Institut, Villigen, Switzerland*

ekaterina.klyushina@sljus.lu.se

We present the results of our recent comprehensive investigation of S=1 honeycomb antiferro- magnet (AFM) BaNi2V2O8, which we show to be an ideal two-dimensional (2D) magnet. Further- more, it has weak planar (XXZ) anisotropy, which makes it a candidate for Berezinsky-Kosterlitz - Thouless behavior. Our study employs a combination of experimental and theoretical techniques. First, we measure and analyze the magnetic excitation spectrum of BaNi2V2O8 at T=4K using the inelastic neutron scattering technique to solve the Hamiltonian of this compound [1]. Then, we explore both temperature regions below and above ordering temperature TN=47.75 K±0.25 K using neutron scattering, muon spectroscopy and magnetic susceptibility measurements [2].

Based on our experimental findings we developed a consistent phase diagram of BaNi2V2O8 (Fig.1) showing that it behaves as a quasi-2D AFM over a wide temperature range displaying a series of crossovers from 2D XY to 2D XXZ and then to 2D Heisenberg behavior with increasing temperature [2]. Moreover, the analysis of the thermal thermal decay of the correlation length reveal that only BKT-theory reproduces the experimental data well over the whole temperature region up 100 K. The Berezinsky-Kosterlitz -Thouless temperature, TBKT=44.70 K ±0.25 K was extracted and founded to be lower than TN, therefore we expect the presence of decoupled spin vortex-antivortex pairs just above TN within the temperature range of TN-TXY where the system behaves as a 2D XY -magnet. Our results are in agreement with the results of Classical and Quantum Monte-Carlo simulations performed for the model Hamiltonian of BaNi2V2O8 [2].

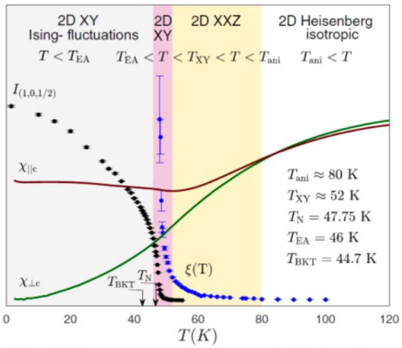


Fig.1 Phase diagram of $BaNi_2V_2O_8$ which is from Ref. [2]

References

[1] E. S. Klyushina, B. Lake, A. T. M. N. Islam, J. T. Park, A. Schneidewind, T. Guidi, E. A. Goremychkin, B. Klemke and M. Månsson . *Phys. Rev. B* **96** 214428 (2017)

[2] E. S. Klyushina, J. Reuther, L. Weber, A. T. M. N. Islam, J. S. Lord, B. Klemke, M. Månsson, S. Wessel, and B. Lake *Phys. Rev. B* **104**, 064402 (2021)

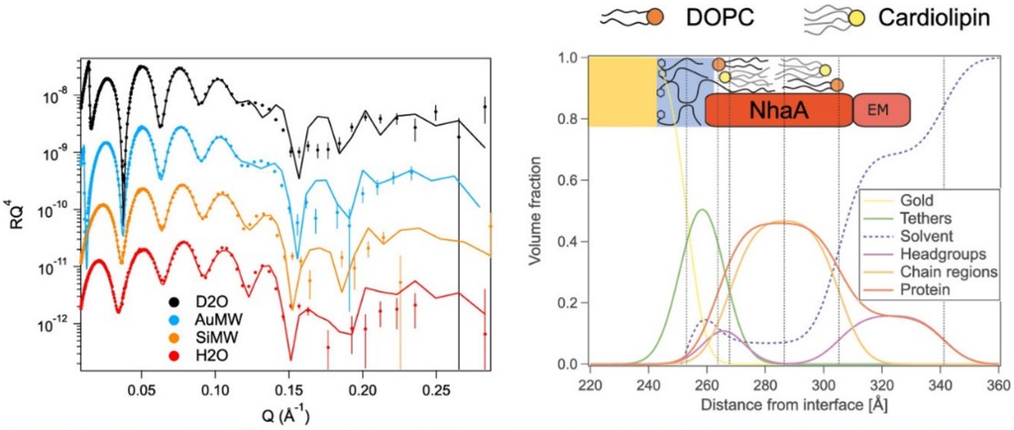
Nanostructural characterization of cardiolipin-containing tethered lipid bilayers for protein incorporation

Sebastian Köhler[a], Giovanna Fragneto[b], Jean-Pierre Alcaraz[c], Andrew Nelson[d], Donald K Martin[c], Marco Maccarini[c]

[a]Lund University, Lund, Sweden
[b]ILL, Grenoble, France
[c]Université Grenoble Alpes, Grenoble, France
[d]ANSTO, Lucas Heights, Australia

sebastian.kohler@linxs.lu.se

The study of active membrane proteins requires an environment which is as close as possible to their natural environment, while at the same time keeping the system as simple as possible to allow for an experimental characterization. Tethered lipid bilayer membranes (tBLMs) represent an experimentally accessible and stable model for biological membranes that offers a high level of control over the structure and can form a more natural environment than the widely used solid supported bilayers. Here we used neutron reflectometry to investigate the structure of tBLMs and to characterize the incorporation of the NhaA sodium proton antiporter protein into the bilayer. We report a study of two different tether architectures using polyethylene glycol linkers - one grafted to a gold surface and one to a silicon surface – which were used to form tBLMs with varying fractions of the lipid cardiolipin. At high fractions cardiolipin caused significant changes in lipid bilayer structure due to interaction with the tethering molecules. NhaA was successfully incorporated into both these tether architectures at high fractions and the modulation of structural properties of the membrane was detailed.



Neutron reflectometry data of a tBLM containing 50% cardiolipin with incorporated NhaA protein. Left: reflectivity curves at different solvent contrasts, right: volume fraction distribution of bilayer system components.

References

[1] Köhler S, Fragneto G, Alcaraz JP, Nelson A, Martin DK, Maccarini M. Nanostructural Characterization of Cardiolipin-Containing Tethered Lipid Bilayers Adsorbed on Gold and Silicon Substrates for Protein Incorporation. *Langmuir*. 2021;37(30):8908-8923. doi:10.1021/acs.langmuir.1c00119

Thermal expansion modeling of mullite-type $Bi_2Fe_4O_9$, $Bi_2Fe_2Mn_2O_{10}$ and $Bi_2Mn_4O_{10}$ across magnetic phase transitions studied by neutron elastic scatterings

M. Mangir Murshed[a,b], *Naveen K. C. Muniraju*[c,d], *Thorsten M. Gesing*[a,b]

[a] *University of Bremen, Institute of Inorganic Chemistry and Crystallography, Leobener Straße 7, D-28359 Bremen, Germany*
[b] *MAPEX Center for Materials and Processes, Bibliothekstraße 1, D-28359 Bremen, Germany*
[c] *Institute of Physics, Bijenička 46, 10000 Zagreb, Croatia*
[d] *AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, 30-059 Krakow, Poland*

murshed@uni-bremen.de

To model the lattice thermal expansion using the Grüneisen approximation below the spin-ordering temperature regime a general problem exists to distinguish the energy contribution between the magnetic and the phonon excitations. Indeed, the magento-phononic coupling is not trivially additive, thus cannot be independently solved. Here we study the lattice thermal expansion of mullite-type $Bi_2Fe_4O_9$, $Bi_2Fe_2Mn_2O_{10}$ and $Bi_2Mn_4O_{10}$ compounds across paramagnetic to antiferromagnetic phase transitions. Temperature-dependent neutron powder diffraction data Rietveld refinement allowed for lattice thermal expansion of the phases. Each metric parameter is modelled using a single Debye term to describe the temperature-dependent internal energy of the Grüneisen first-order approximation [1]. The Debye temperatures are calculated from the evolution of both metric and isotropic displacement parameters. In each case, due to strong magneto-volume effect the Debye-fit significantly deviate from that of the observed metric parameter below the respective Néel temperatures. The antiferromagnetic orderings are calculated as 39(1) K, 20(2) K and 40(1) K, respectively, for $Bi_2Fe_4O_9$, $Bi_2Fe_2Mn_2O_{10}$ and $Bi_2Mn_4O_{10}$ from the Landau fitting of the reduced cell volume. The magneto-volume coupling constants are calculated by plotting the reduced volume versus the squared bulk magnetization per magnetic cation, showing almost a linear relationship. We also tested a transition function attached to Debye-Einstein-Anharmonicity model [1,2], describing the lattice thermal expansion behaviors across the magnetic/nuclear phase transitions.

References

[1] M. M. Murshed, P.Zhao, A. Huq, Th. M. Gesing, Z. Allg. Anorg. Chem. 644 (2018) 253-259.
[2] M. M. Murshed, M. Šehović, M. Fischer, A. Senyshyn, H. Schneider, Th. M. Gesing, J. Am. Ceram. Soc. 100 (2017) 5259-5273.

Study of phase equilibria of novel deep eutectic solvents systems

Sushma P. Ijardar[a]

[a]*Department of Chemistry, Veer Narmad South Gujarat University, Surat 395007, India*

sijardar@vnsgu.ac.in

The quest for green solvents that can be substituted for volatile organic compounds (VOCs) and somehow toxic ionic liquids (ILs) has led to the discovery of the 21st century green solvents known as deep eutectic solvents (DESs)]. Due to their green nature, low cost, tuneability, high biodegradability and ease of preparation compared to other solvents, DESs are now rapidly emerging in several applications [1,2] . In this work, aqueous biphasic systems (ABSs) formed by different DESs and inorganic salts at ambient temperature were investigated [4]. The DESs were synthesized using benzyltrimethyl ammonium chloride, benzyltriethyl ammonium chloride, and benzyltributyl ammonium chloride as the hydrogen bond acceptors (HBAs) combined with PEG of various molar mass as hydrogen bond donors (HBDs) [3] . The synthesized DESs were used to form ABS with some inorganic salts using cloud point titration method. The characterization of DES in DES rich phase was characterized using SANS. Empirical nonlinear expression developed by Marchuk et al. was used to construct the binodal curves while the tie lines were determined using Othmer-Tobias equations [4]. Phase-forming capacity of the phosphate salts were studied alongside relevant factors affecting phase formation [5].

References

[1] E.L. Smith, A.P. Abbott, K.S. Ryder, Chem. Rev. (2014) 114, 11060–11082.
[2] Q. Zhang, K.D.O. Vigier, S. Royer, F. Jerome, Chem. Soc. Rev. (2012) 41, 7108–7146.
[3] S.P. Ijardar, J. Chem. Thermodyn. 140 (2020), 105897
[4] M.G. Freire, A.F.M. Claudio, J.M.M. Araujo, J.A.P. Coutinho, I.M. Marrucho, J.N. C. Lopes, L.P.N. Rebelo, Chem. Soc. Rev. 41 (2012) 4966–4995
[5] E. A. Oke, S. P. Ijardar, Biochem. Eng. J.176 (2021) 108211

Towards measuring surface dynamics with neutrons

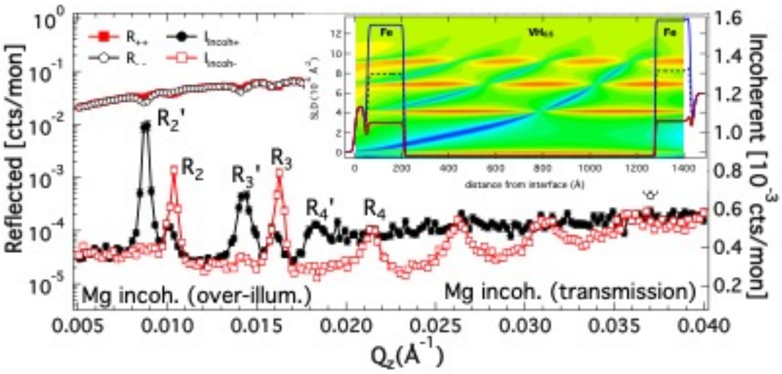
Max Wolff[a], Adrien Perrichon[b], Maths Karlsson[c], Anton Devishvili[d], Boris Toperverg[e]

[a]Department for Physics and Astronomy, Uppsala University, Uppsala, Sweden
[b]ISIS Facility, STFC Rutherford Appleton Laboratory, Didcot, UK
[c]Chalmers University of Technology, Gothenburg, Sweden
[d]Institute Laue Langevin, Grenoble, France
[e]Ruhr-University Bochum, Bochum, Germany

max.wolff@physics.uu.se

The high penetration of neutrons allows the study of buried interfaces and the energy of cold neutrons makes them an excellent probe for dynamics. However, the limited brilliance of neutron sources makes the combination of reflectometry and spectroscopy challenging. This is especially true for materials containing hydrogen, which has a large incoherent scattering cross section allowing to probe tracer diffusion but often treated as background in low Q studies. Even though surface science has advanced enormously, many scientific questions that may be addressed by neutron spectroscopy remain unresolved. Examples are spin wave dispersions, dynamics of glass formers and self-assembled monolayers as well as the dynamics of water close to membranes and the dynamics of lithium and protons in thin films in energy storage or conversion materials.

In this presentation we show how resonant effects can be used to quantify any scattering from thin films and interfaces. A consistent evaluation of the neutron reflectivity at conditions of total external reflection together with the known scattering cross section allows to identify scattering [1]. It turns out that a significant fraction of the incident beam, on the order of 10-20%, may get scattered incoherently in a 100 nm thick vanadium hydride film [2], thus paving the road towards neutron spectroscopy of thin films. The figure below (insert) shows the scattering length density (SLD) profile for neutrons impinging under grazing incidence onto a 100 nm thick vanadium hydride resonator sandwiched between iron layers. Resonances are visible for specific incident wave numbers. At these resonances, dips are present in the reflectivity (main panel) and neutrons are registered by a detector mounted behind the sample. This demonstrates that the dips in reflectivity originate from enhanced incoherent scattering, mainly from hydrogen.



References

[1] A. Perrichon et al., Phys. Rev. B 103, 235432 (2021).
[2] M. Wolff et al., Phys. Rev. Lett. **123**, 016101 (2019).

Abstract: 063

Longitudinal conical magnetic structure in scandium doped M-type Barium hexaferrite

Surbhi Gupta[a], V. Siruguri[b], K. G. Suresh[a]

[a]Department of Physics, Indian Institute of Technology Bombay, Powai, Mumbai 400076
[b]UGC-DAE Consortium for Scientific Research Mumbai Centre, BARC Campus, Mumbai 400085, India

surbhi1990@iitb.ac.in

M-type barium hexaferrites (BaM) has attract a lot of attention due to their multiferroic and other functional properties [1, 2]. We have investigated the doped M-Type Barium hexaferrite, BaFe12- xTxO19, where T = Sc with x = 2.5. The structural and magnetic properties were characterized using XRD, VSM and Neutron diffraction measurements. XRD analysis reveals that the samples are in single phase with space group P63/mmc. Magnetization data reveal interesting behavior, Zero- field cooling (ZFC) and field cooled warming (FCW) curves in temperature range of 5 K to 750 K indicate several transitions for the Scandium doped compound. Transitions observed at lower temperatures indicate antiferromagnetic order. Temperature dependence neutron diffraction measurements performed at a wavelength of, λ = 2.315 Å, in the temperature range 3 K – 300 K, analysis of neutron data reveals non-collinear magnetic order at the lowest temperature. Magnetic satellite reflections start appearing at low angles on decreasing of temperature, refinement of this magnetic reflection indicates the presence of conical magnetic structures at low temperatures. This shows that the direction of magnetic moments, when compared with the parent compound, is no longer along the hexagonal c-axis. Magnetic structures for the Scandium doped with doping concentration x = 2.5 is analyzed and presented in details.

References

[1] R. C. Pullar Prog. Mater. Sci. 57 1191 (2012)
[2] A. Moitra, S. Kim, S. G. Kim, S. C. Erwin, Y. K. Hong and J. Park, Comp. Condens. Matter 1 45 (2014).

Investigations of boron carbide based neutron absorption layers for an early-stage superconducting neutron detector

Mette Bybjerg Brock[a], *Asger Bech Abrahamsen*[b], *Anders C. Wulff*[c], *Matteo Busi*[d], *Luise Theil Kuhn*[a]

[a]*DTU Energy, Technical University of Denmark, Kgs. Lyngby, Denmark*
[b]*DTU Wind Energy Systems, Roskilde, Denmark*
[c]*SUBRA A/S, Farum, Denmark*
[d]*Paul Scherrer Institut, Villigen, Switzerland*

mebyb@dtu.dk

An increasing demand for monitoring neutrons in high neutron flux environments has led to investigations into many new types of detectors. These include the use of a variety of different neutron sensitive materials as well as different detection principles from gas to completely solid-state. A superconducting detector has been investigated for high-flux environments.

For this early-stage detector, neutron response tests have been carried out at the Paul Scherrer Institute in Switzerland. Key results of these tests are to be presented along with models investigating the limitations and possibilities of this technology.

The authors wish to thank Chung-Chuan Lai and Linda Robinson from the ESS Detector Coatings Workshop, Linköping, SE, for the deposition of the absorption layer.

Polarized SANS and GISANS: research and data analysis at LU/ESS

Annika Stellhorn[a,b], Wai Tung Lee[b], Emmanuel Kentzinger[c], Oskar Stepancić[a], Lingjia Shen[a], Elizabeth Blackburn[a]

[a]Lund University, Division of Synchrotron Radiation Research, 22100 Lund, Sweden
[b]European Spallation Source (ESS), Instrument Technologies Division, 22484 Lund, Sweden
[c]Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS-2) and Peter Grünberg Institut (PGI-4), JARA-FIT, 52428 Juelich, Germany

annika.stellhorn@sljus.lu.se

Small-Angle-Neutron-Scattering (SANS) and Grazing-Incidence-SANS (GISANS) techniques have seen a remarkable growth in their application in magnetic material studies. One example for this is condensed-matter system with complex spin textures such as magnetic chirality, which offers great potential for applications in spintronic devices. Together with neutron polarization analysis, *polarized* SANS investigates systems with magnetic periods on the mesoscopic scale, being complemented by its surface-sensitive counterpart, polarized GISANS. These techniques rely on proper data reduction and analysis to extract the physics.

My research focuses on using and establishing suitable data reduction and analysis methods in these techniques. First, our research on magnetic chiral structures occurring in magnetoelectric single crystal Ba2-xSrxMg2Fe12O22 [1], and in magnetic-domain-hosting compound FePd thin film will be presented. Second, magnetic field simulations of polarized GISANS setups, to optimize the neutron spin-transport, will be discussed. Third, current developmental efforts on polarized SANS data analysis in software SASView [2] will be reported. Polarization analysis has been incorporated as an integral part of the European Spallation Source instrument suite [3]. An introduction of its current scope will also be provided.

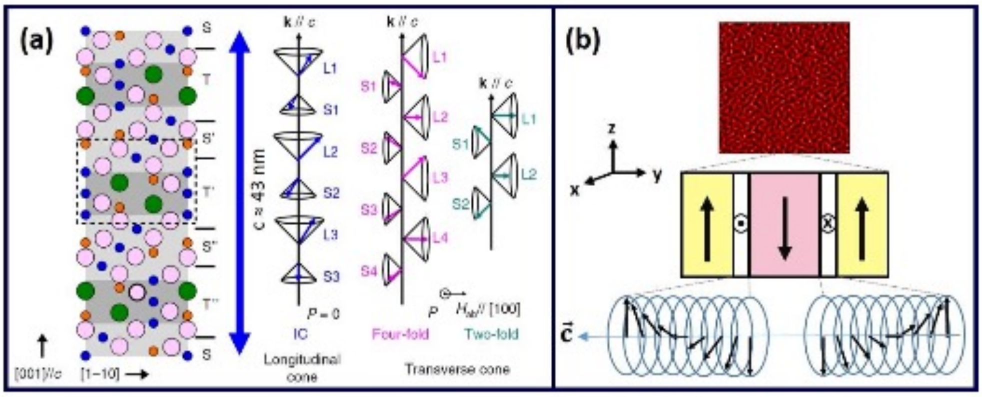


Figure 1 (a): chiral magnetic spin structures in magnetoelectric $Ba_{2-x}Sr_xMg_2Fe_{12}O_{22}$ [1]. (b) Ferromagnetic FePd thin films with perpendicular magnetic anisotropy, forming a maze domain structure and chiral Bloch domain walls.

References

[1] K. Zhai *et al.*, Nature Communications **8**, 519 (2017).
[2] www.sasview.org
[3] W. T. Lee *et. al.*, Report on ESS Polarisation Workshop, ESS-3549713 (2020).

Quantum cascade laser-based infrared spectrophotometer for the combination with small angle neutron scattering for life science applications.

Seyed Mohammad Mahdi Dadfar[a], *Aurel Radulescu*[b], *Andreas Stadler*[c], *Jörg Fitter*[a], *Tobias E. Schrader*[b]

[a]*Physikalisches Institut (IA), AG Biophysik, RWTH Aachen, Aachen, Germany.*
[b]*Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Garching, Germany.*
[c]*Jülich Center for Neutron Science (JCNS-1) and Institute for Biological Information Processing (IBI-8), Forschungszentrum Jülich, Jülich, Germany.*

mahdi.dadfar@rwth-aachen.de

Monitoring the amide I band, infrared spectroscopy can give information on the fold of the protein and also allows to follow aggregation phenomena. Small angle neutron scattering also reports on the global structure of proteins in solution and can give information on the shape of growing aggregates or folded proteins in solution.

In the framework of the BMBF-funded project "Neut-IR", we would like to explore the capabilities of quantum cascade lasers (QCLs) for the combination of infrared spectroscopy with small angle neutron scattering. The advantages of QCL based IR spectrophotometers are superior Gaussian beam characteristics and higher time resolution (1 second) due to a higher spectral density as compared to the glow bar infrared light sources of the Fourier-transform infrared spectrometer (FTIR). Their disadvantage is the more complicated pulsed mode of operation and the limited spectral width they can cover.

As scientific samples, the effect of a pH drop on protein aggregation and amyloid like structure formation in insulin or in Poly-L-Glutamic Acid (PLE) is investigated. In this contribution we focus on insulin. Insulin is a peptide hormone, secreted in ß-cells of the pancreatic islets. Insulin was dissolved in a phosphate buffer, where the pH was adjusted to 2. At room temperature the sample was pumped through varying combinations of flow through cells of the FTIR spectrometer, the QCL, the UV-Visible spectrophotometer and the static light scattering device. Thereby we could follow the amyloid like structure formation on the very same sample using many different techniques in parallel. The initial result related to the amyloid like structure formation, obtained by FTIR and QCL measurements is shown in Figure 1. As seen, in both cases the amyloid formation peak can be observed at 1627 cm^{-1}. Furthermore, a peak near 1550 cm^{-1} is due to the amide I vibration.

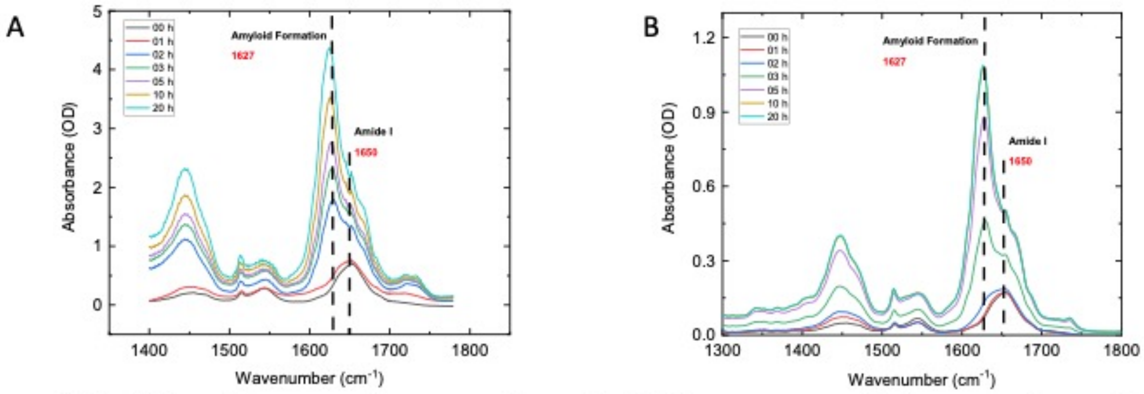


Figure 1: (A) QCL based IR spectrophotometer (B) standard FTIR measurement for human insulin at pH = 2 with a phosphate buffer of 200 mM monitored over several hours.

Probing double exchange spin dynamics within valence delocalised magnetic molecules using various spectroscopic techniques

R. T. Christiansen[a,b], S. Wu[c], O. Sato[c], R. Clérac[d], J. Ollivier[a], M. L. Baker[b]

[a]Institut Laue-Langevin, F-38042 Grenoble Cedex 9, France
[b]Department of Chemistry, The University of Manchester, Manchester M13 9PL, UK
[c]Institute for Materials Chemistry and Engineering & IRCCS, Kyushu University, Fukuoka 819-0395, Japan,
[d]CNRS, UMR 5031, Centre de Recherche Paul Pascal (CRPP), F-33600 Pessac, France

christiansen@ill.fr

In molecules containing mixed-valence (MV) metal ions, delocalisation of one or more spin-polarised valence electrons can lead to ferromagnetic exchange couplings between metal centres; a phenomenon known as double exchange (DE)[1]. It is fundamentally different from traditional Heisenberg exchange (HE) since DE eigen-energies scale linearly rather than quadratically with the total spin. DE can be harnessed to engineer promising molecules for quantum technologies[2], and MV molecules play a fundamental role as electron transfer active sites in enzymes[3].

Here, we present studies on two types of molecules exhibiting DE: A new series of high-spin (HS) FeII-FeIII dimers with slight variations in the bridge, and a polynuclear molecule (abbr. Fe14) with a $S = 14$ ground state which formally contains 4 HS FeIII, 2 HS FeII and 6 low-spin FeII[4]. Magnetometry confirmed that DE leads to a $S = 9/2$ ground state in the dimers (Fig. 1, left), while EPR and UV-vis showed that perturbing the bridge affects e- delocalisation and zero-field splitting. For Fe14, powder neutron spectroscopy revealed spin state energies non-compliant with a HE model and that the excited states are unchanged in applied E-fields of 0-3 kV/mm (Fig. 1, right).

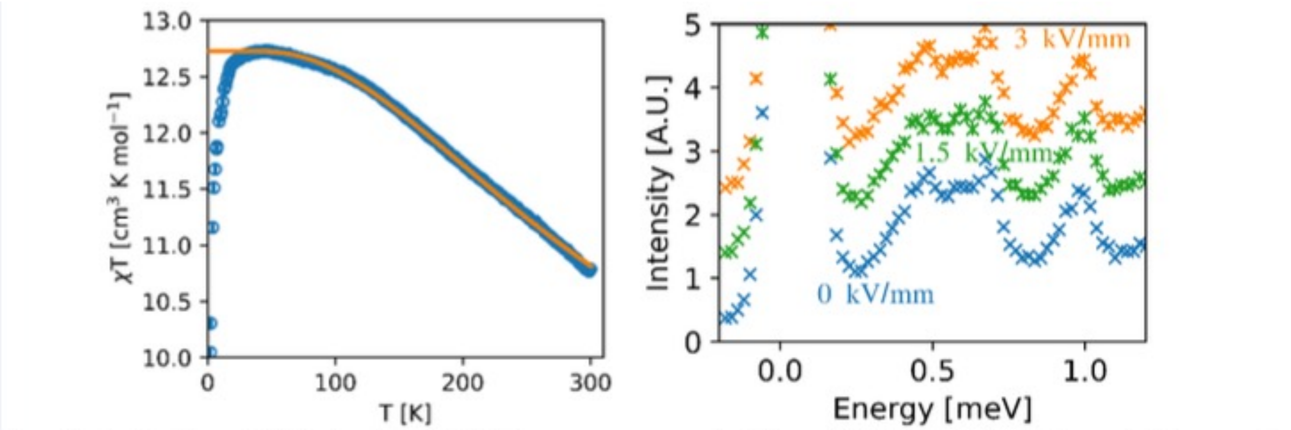


Fig. 1. Left: χT vs. T (blue) of Fe^{II}-Fe^{III} dimers measured at H = 1000 Oe with a DE model (orange) fitted to data. The decrease at low T is due to ZFS not accounted for in the model. **Right:** INS spectra of Fe_{14} at various applied E-fields from LET@ISIS (Ei = 1.8 meV, T = 1.8 K), offset for clarity.

References

[1] C. Zener, *Phys. Rev.*, 82, 403 (1951)
[2] A. Palii et al, *Coord. Chem. Revs.*, 426, 213555 (2021)
[3] S. Sharma et al, *Nat. Chem.*, 6, 927 (2014)
[4] W. Huang, S. Wu, ..., O. Sato et al, *Nat. Commun.*, 10, 5510 (2019)

The role of intramembrane peptide nanoaggregates in bacterial fast-killing

Mingrui Liao,[a] Haoning Gong,[a] Xuebo Quan,[b] Xuzhi Hu,[a] Zheng Chen,[b] and Jian R. Lu,[a]

[a]Biological Physics Laboratory, Department of Physics and Astronomy, School of Natural Science, The University of Manchester, Oxford Road, Manchester M13 9PL, UK
[b]School of Chemistry and Chemical Engineering, Guangdong Provincial Key Laboratory for Green Chemical Product Technology, South China University of Technology, Guangzhou 510640, China.

J.lu@manchester.ac.uk

Recent development of antimicrobial peptides (AMPs) has focused on the rational design of short sequences with less than 20 amino acids due to their relatively low synthesis costs and ease of correlating structure-function relationship. However, there remain gaps in our understanding of how these short cationic AMPs interact with bacterial outer and inner membranes and how these interactions underline their antimicrobial efficacy and dynamic bacterial killing.[1] In this work, we have examined the membrane-lytic actions of three synthetic AMPs including $G(IIKK)_3I\text{-}NH_2$ (G_3), $G(IIKK)_4I\text{-}NH_2$ (G_4) and $GLLDLLKLLLKAAG\text{-}NH_2$ (LDKA) and a natural AMP $GIGAVLKVLTTGLPALISWIKRKR\text{-}NH_2$ (Melittin). The mechanistic processes of membrane damage and disruption strength of the four AMPs were characterized by both experimental measurements and molecular dynamics simulations. The combined studies revealed that G_3 and G_4 had higher antimicrobial efficacy and better membrane selectivity than Melittin and LDKA. Their distinctive antimicrobial actions arose from the dual actions of strong binding to outer and inner membranes and formation of intramembrane nanoaggregates.[2-3] Furthermore, intramembrane binding and aggregation from all AMPs studied intensified as the ratio of peptide to lipid increased, but the most effective membrane insertion of G_3 was well in line with its most efficient antimicrobial action.

References

[1] D. Ciumac; H. Gong; X. Hu; J. R. Lu, Membrane targeting cationic antimicrobial peptides. *J Colloid Interface Sci* **2019,** *537*, 163-185.
[2] R. M. Epand; R. F. Epand, Bacterial membrane lipids in the action of antimicrobial agents. *J Pept Sci* **2011,** *17* (5), 298-305.
[3] D. Lopez; J. D. Nickels; S. Chatterjee; C. B. Stanley; S. Qian; X. Cheng; D. A. A. Myles; R. F. Standaert; J. G. Elkins; J. Katsaras, The in vivo structure of biological membranes and evidence for lipid domains. *PLOS Biology* **2017,** *15* (5), e2002214.

Abstract: 073

Combining SANS, Cryo-EM and computational modeling to reveal structure of Hepatitis B virus

Wojciech Potrzebowski[a,b], Najet Mahmoudi[c], Ingemar André[b]….

[a]European Spallation Source ERIC, Lund, Sweden
[b]Department of Biochemistry and Structural Biology, Lund University, Lund, Sweden
[c]STFC, Rutherford-Appleton Labs, Didcot, UK

wojciech.potrzebowski@ess.eu

The genetic material of viruses is typically protected in an icosahedral capsid, which is primarily assembled from over a hundred subunits of the same protein in a spontaneous self-assembly process. Due to the dynamic nature of self-assembly it is difficult to determine the interaction of capsid protein and nucleic acid even at the fully assembled state. Our research aims to provide a detailed understanding of how the RNA is incorporated into the capsid of Hepatitis B virus (HBV) and how the overall structure of capsid is affected by the presence of RNA. In order to address these questions, we developed a method combining contrast variation SANS, Cryo-EM and computational modelling. We used Bayesian statistical modeling to decompose profiles of protein and RNA from the contrast variation data. One of the advantages of Bayesian framework is the possibility of incorporating prior information (data from another SANS experiment) to guide inference process. However, from Cryo-EM we generated radial profiles, which were used as constraints in modeling of SANS data. The decomposed SANS profiles together with Cryo-EM radial profiles were subsequently used in SasView [1] to fit coarse-grained models. Based on the inferred structural parameters we were able to draw conclusions regarding protein and RNA interactions in virus capsid.

References

[1] SasView version 5.0.4, https://doi.org/10.5281/zenodo.4467703

Combined INS, FT-IR and DFT evidence of H_2 induced ductility in a Pt/Al_2O_3 catalyst

Andrea Piovano,[a] Eleonora Vottero,[b] Alberto Ricchebuono,[b] Monica Jiménez-Ruiz,[a] Riccardo Pellegrini,[c] Céline Chizallet,[d] Pascal Raybaud,[d] Elena Groppo.[b]

[a] Institut Laue-Langevin (ILL), 38042 Grenoble, France
[b] Department of Chemistry, INSTM and NIS Centre, University of Torino, I-10135 Torino, Italy
[c] Chimet SpA - Catalyst Division, I-52041 Viciomaggio Arezzo, Italy
[d] IFP Energies nouvelles, BP3-69360 Solaize, France

piovano@ill.fr

Supported platinum nanoparticles are largely employed in industrial hydrogenation processes thanks to their great catalytic activity toward H_2 splitting. However, their ability to activate and split the H_2 molecules at the metal surface to form reactive hydride species under reaction conditions is still matter of debate and subject of extensive research effort.

The sample, prepared by the industrial partner Chimet S.p.A, is a 5% wt Pt nanoparticle catalysts supported on a high surface area transition alumina.

Inelastic Neutron Scattering (INS) spectroscopy had been a fundamental probe, thanks to its great sensitivity towards the vibrational modes of all H-containing species. The INS spectra of the sample were measured under a high and low H_2-pressure on the IN1-Lagrange instrument at the ILL, resulting in significantly different spectral profiles [1]. The spectra were interpreted on the basis of an extensive DFT simulation work comprehensive of 26 different $Pt_xH_y/\gamma Al_2O_3$ models, performed in collaboration with IFPEN. The comparison clearly indicated markedly different morphologies and interaction with the support. The spillover of some H onto the support and the following formation of surface -OH groups was also observed. Complementary information was obtained through FT-IR experiment performed in operando conditions. The evolution of the system upon the progressive decrease of the H_2 concentration could be followed and the spectral evolution clearly indicated the conversion of multi-folded Pt-H species into linear ones [2]. Both these findings are in good agreement with previous AIMD simulations, predicting the reconstruction of Pt_{13} nanoparticles on γ-Al2O3 as a function of the temperature and the H_2 equilibrium pressure [3].

Overall, this study provided important new complementary information on the ductile behavior of Pt/Al_2O_3 catalyst under hydrogenation reaction conditions, representing a new important step in the understanding of their real nature.

References

[1] E. Vottero et al, "Evidence for H_2-Induced Ductility in a Pt/Al_2O_3 Catalyst", ACS Catal. 2022, 12, 10, 5979–5989

[2] M. Carosso et al, "Dynamics of Reactive Species and Reactant-Induced Reconstruction of Pt Clusters in Pt/Al_2O_3 Catalysts", ACS Catalysis, 2019, 9, 8, 7124-7136

[3] C. Mager-Maury et al, "H_2-Induced Reconstruction of Supported Pt Clusters: Metal-Support Interaction versus Surface Hydride." ChemCatChem. 2011, 3, 1, 200−207

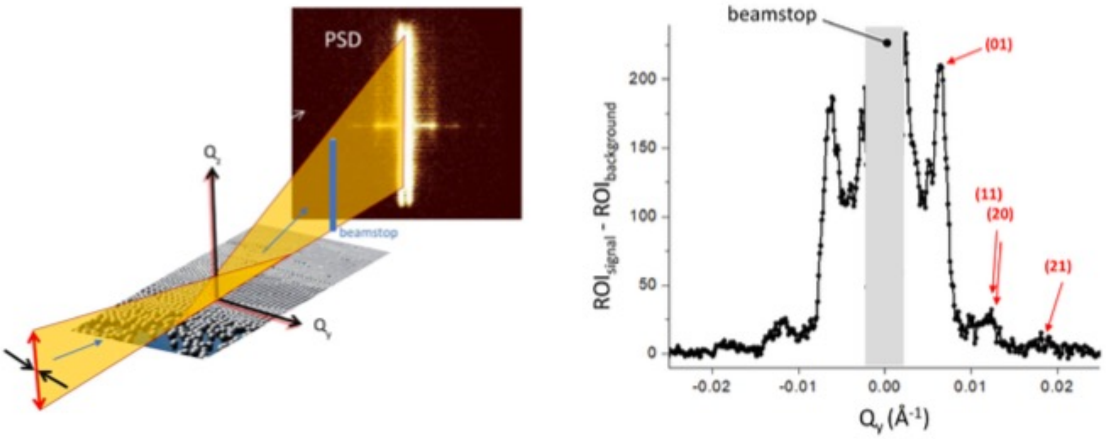
Flux enhancement for GISANS studies using fan shaped beams

Alexei Vorobiev[a,b], Nicolò Paracini[c], Marité Cárdenas[c], Max Wolff[a]

[a]Department for Physics and Astronomy, Uppsala University, Uppsala,
[b]Institute Laue-Langevin, Grenoble, France,
[c]Department of Biomedical Science and Biofilm Research Center for Biointerfaces, Malmö University, Malmö, Sweden

alexey.vorobiev@physics.uu.se

We show that one-dimensional GISANS measurements can be done using a divergent incident neutron beam and integrating the detector images along the out-of-plane direction. In this way we get a significant increase in incident-beam intensity but compromise on depth resolution. For single films and interfaces this is acceptable. We still take benefit of the advantages of neutrons, which are sensitive to isotopic contrast and the magnetic induction. The method also allows straight forward investigations of buried interfaces which cannot be achieved with any microscopy based method. We show that this method works well on the monochromatic, high resolution, neutron reflectometer Super ADAM [1] and resolve the lateral structure of iron inclusions in an aluminium oxide matrix. In addition, we resolve the particle form factor of silica spheres as well as the structure factor from their hexagonal arrangement on a silicon as well as water substrate [2]



Left: Sketch of the scattering geometry for one-dimensional GISANS studies. Right: Vertically integrated GISANS signal from 100 nm silica particles on water surface corrected for the background and plotted versus Q_y on a linear scale.

References

[1] Vorobiev, A., Devishvili, A., Palsson, G., Rundlof, H., Olsson, A., Dennison, A., Wolff, M., Giroud, B., Aguettaz, O., Hjörvarsson, B. Recent upgrade of the polarized neutron reflectometer Super ADAM. Neutron News 26(3), 25 (2015)

[2] VorobievA.,ParaciniN.,CardenasM.,WolffM.,Π-GISANS:probinglateralstructureswithafan shaped beam. Scientific Reports | (2021) 11:17786 | https://doi.org/10.1038/s41598-021-97112-x

Abstract: 078

Effect of surface charges on structure and interactions of soft colloids

Lingsam Tea[a], Jörg Stellbrink[a] and Lutz Willner[a]

[a]JCNS-1, Forschungszentrum Jülich GmbH 52425 Jülich

j.stellbrink@fz-juelich.de

Soft colloids are ubiquitous in synthetic and biological material as e.g., vesicles, dendrimers, microgels, polymer-grafted nanoparticles, micelles, star polymers and certain proteins. They display a dual character between a polymer and the archetypical hard sphere colloid. Due to this hybrid nature, soft colloids macroscopically show interesting structural [1] and dynamic properties [2]. Flow behavior of crowded solutions, (visco-) elastic properties of jammed states as gels and glasses and finally crystal structure and stability of other highly ordered phases crucially depend on “softness” [3].

We established micelles formed by amphiphilic block copolymers as an easy and elegant model system to tailor colloidal softness [4]. Parameters to tailor the “softness” start on a microscopic level by selecting the chemical structure of the hydrophobic block, by varying the solvophobic-to-solvophilic block ratio, the absolute block molecular weights, polydispersity of the hydrophobic block, and/or the interfacial tension, core crystallinity etc.

In the present work, we introduce additional surface charges on the micelles, which will result in an electrostatic repulsion. The charges are implemented by oxidation of only the terminal hydroxy group of the PEO block into carboxy group by Bobbitt’s salt [5]. The carboxy function being a weak acid, the number of charges on the particles can be tuned by changing the pH or by the ionic strength. Therefore, range and strength of electrostatic repulsion relative to the inherent steric repulsion due to the micellar corona can be precisely adjusted.

We studied intra- and interparticle structure over a broad concentration range by SANS as a function of maximum charge (i.e. aggregation number) but also as a function of effective charges (adjusted by pH and ionic strength/salt concentration). Form factors are analyzed with a core–shell model that has been used in an earlier study for uncharged micelles [3]. It is shown that single micellar properties remained the same independently of the number of surface charges. However, for charged systems the structure factor is visible even at very dilute concentration denoting the long-range electrostatic repulsion dominating in the liquid phase. Structure factors are analyzed either by Hard Sphere (Percus-Yevick), ultra-soft (Likos-Löwen) and/or a modified Hayter-Penfold models [6]. The experimental phase diagram revealed that by increasing the concentration, a liquid-crystal transition occurs for all systems, but the electrostatic interaction leads to a liquid - solid phase transition at a much lower concentration compared to the uncharged micelles.

References

[1] S. Gupta, M. Camargo, J. Stellbrink, J. Allgaier, A. Radulescu et al., Nanoscale **7** (2015) 13924.
[2] S. Gupta, J. Stellbrink, E. Zaccarelli, C. N. Likos et al. Physical Review Letters **115** (2015) 128302.
[3] M. Amann, L. Willner, J. Stellbrink, A, Radulescu, and D. Richter, Softmatter **11** (2015) 4208.
[4] M. Laurati, J. Stellbrink, R. Lund, L. Willner, et al. Physical Review Letters **94** (2005) 195504.
[5] Bobbitt et al., Organic Reactions, (NY) **74** (2010) 103.
[6] M. Heinen, P. Holmqvist, A. J. Banchio and G. Nägele, J. Chem. Phys. **134** (2011) 044532.

Neutron structure of *Leishmania mexicana* triosephosphate isomerase variant E97Q reveals a possible general acid

Swati Aggarwal[a], Justin Bergmann[b], Vinardas Kelpsas[c], Octav Caldararu[b], Matthew P. Blakeley[d], Nicolas Coquelle[d], Rikkert Wierenga[e], Claes von Wachenfeldt[c], Ulf Ryde[b], Esko Oksanen[a]

[a]European Spallation Source ESS ERIC, Lund, Sweden
[b] Theoretical Chemistry, Lund University, Lund, Sweden
[c] Biology, Lund University, Lund, Sweden
[d] Institut Laue-Langevin, Grenoble, France
[e] Biochemistry, University of Oulu, Oulu, Finland

esko.oksanen@ess.eu

Triosephosphate isomerase (TIM) is a key enzyme in glycolysis that catalyses the interconversion of glyceraldehyde- 3-phosphate (GAP) and dihydroxyacetone phosphate (DHAP). This simple reaction involves the shuttling of protons mediated by protolysable side chains. The catalytic power of TIM is thought to stem from the ability to facilitate the deprotonation of a carbon next to a carbonyl group to generate an enediolate intermediate. The enediolate intermediate is well mimicked by the inhibitor 2-phosphoglycolate (PGA) and a previous neutron structure [1] showed that E167 was protonated, confirming it as the general base [2]. The identity of the general acid catalyst remains debated. One candidate is K10, where we saw potential indications of deprotonation, but finally concluded that K10 was not deprotonated. To further explore this we mutated E97 that hydrogen bonds to K10 and determined the neutron structure the variant E97Q. Surprisingly the mutation leads to a conformational change in K10 and the inhibitor, explaining a previously observed minority conformation of the inhibitor. We then performed joint neutron-X-ray refinement followed by quantum refinement, but the quantum refinement [3] failed to converge. The reason to this is that K10 is ~30% deprotonated, which is very challenging to accurately model in quantum chemistry methods. This deprotonation of K10 suggests that it could act as a general acid in at least one direction of the reaction. The inhibitor conformation also suggests that the reaction may involve a conformational change of the substrate that has not been previously postulated.

References

[1] Kelpsas et al. (2019) *Acta Cryst.* D**75**, 260-269
[2] Kelpsas et al. (2021) *IUCrJ* **8**, 633-634
[3] Caldararu et al. (2020) *Acta Cryst.* D**75**, 368-380

Abstract: 080

The effect of framework structure/composition on molecular behaviour and controlled drug release from zeolites

Dr Alexander O'Malley[a], *Sandra McHugh*[a], *Dr Markus Appel*[b]

[a]*University of Bath, Bath, UK*

[b]*ILL, Grenoble, France*

a.o'malley@bath.ac.uk

The controlled release of anticancer drug 5-Fluorouracil (5FU) from a zeolite based drug delivery system allows for improved drug bioavailability and helps to prevent premature drug degradation whilst often potentiating its effect, and reducing side effects [1]. Zeolites are crystalline and have tuneable porous channels of molecular dimensions suitable for the accommodation of different drug molecules, and their subsequent controlled release. Of particular importance in terms of precisely controlling a release rate is the zeolite Si/Al ratio, which dictates the number of adsorption sites and has an effect on the steric hindrance of the drug within the zeolite pores, therefore having a significant effect on the controlled drug release profile. QENS was used in combination with molecular dynamics (MD) and Density Functional Theory (DFT) simulations to determine the effect of Si/Al ratio and framework structure (i.e. pore size) on the dynamics of the drug in the pores of zeolite Beta. This allows for an informed correlation between the zeolite characteristics and the controlled release profile of the drug based on its molecular behaviour upon confinement. QENS probed 5FU dynamics in H-Beta with a Si/Al ratio of 19 and 180 using the IN16B instrument (ILL). Confined translational diffusion of 5FU within the zeolite pores was observed, with the obtained EISF fitting to the Volino model of confined spherical motion within a sphere of radius ~6 Å, roughly the size of the intersection between BEA channels, indicating the drug resides here, Fig 1(b). A mobile population of drug molecules of only 6 % in H-Beta (Si/Al = 19) and 4 % in H-Beta (Si/Al = 180), shown in Fig 1(a) indicates the majority of the drug is immobile in the zeolite pores on the instrumental timescale, and a relationship exists between zeolite composition and mobility. Confined diffusion coefficients extracted from line broadenings show similar diffusivities between systems on the order of 2 × 10-10 m2s-1. MD simulations of the systems reproduced QENS observables (EISF and QENS broadenings) to good agreement with the experimental data. The study highlights the effect of zeolite structure and composition on the behaviour of confined drug molecules, rationalizing observed trends between zeolite characteristics and observed controlled release profiles of potential anticancer dosage forms.

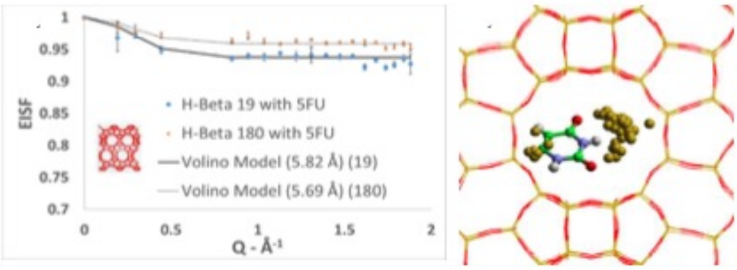


Figure 1. Models of confined rotation (Volino model) fit to experimental EISF of 5-FU in HBEA-19 and 180 and (b) trajectory plot of 5-FU in siliceous BEA at showing confined rotation within a sphere of radius ~6 Å

References

[1] L. Bacakova, M. Vandrovcova, I. Kopova and I. Jirka, Biomater. Sci., 2018, 6, 974–989.

Abstract: 083

Tail-unsaturation enables fine-tuning on the rheology of sugar-based surfactants

Jia-Fei Poon,[a,b] Adrian Sanchez-Fernandez,[b] Anna Leung,[a] Marie Wahlgren[b]

[a]DEMAX group, Scientific Activities Division, European Spallation Source, Lund, Sweden

[b]Food Technology, Nutrition and Engineering, Lund University, Lund, Sweden

Jia-fei.poon@ess.eu

Sugar-based surfactants are considered as green surfactants and widely used in various applications ranged from viscosity modifier to pharmaceutical applications. However, only a scarce number of examples were reported to exhibit non-Newtonian rheological behaviour. Our recent study in hexadecylmaltoside (C16G2) shed light on this area by showing how the headgroup anomeric configuration, axial (□) or equatorial (□) strongly influences micelle formation and structure.1 Small-angle neutron and X-ray scattering revealed that □-C16G2 forms short cylindrical micelles (contour length ca. 1000 Å), whilst □-C16G2 assembles into very long worm-like micelles (contour length ca. 10000 Å). This was attributed to the headgroup interactions, where the □ configuration prompts a closer monomer-monomer packing as induced by hydrophobic and hydrogen bond interactions.

Herein, we report on introduction of double bond to the aliphatic tail influences the self-assembly and macroscopic response of the system, *i.e.* the rheology. Importantly, we showed that both configuration and position of the unsaturation can be utilized to fine-tune the rheological behaviour of sugar-based surfactants (**Figure 1**). Understanding the relationship between the physical properties and the chemical structure of these sugar-based surfactants would provide us the knowledge to tailor the properties of these green surfactants for different applications.

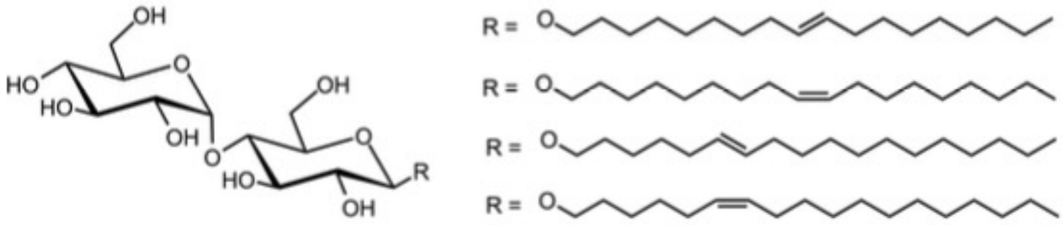


Figure 1. Trans- and cis- configured mono-unsaturated analogues derived from β-$C_{18}G_2$

Reference

[1] Larsson, J.; Sanchez-Fernandez, A.; Leung, A. E.; Schweins, R.; Wu, B.; Nylander, T.; Ulvenlund, S.; Wahlgren. M, *J. Colloid Interface Sci.* **2021**, 581, 895–904.

Multimodal tools to resolve assembly mechanisms in the α –> β transition of recombinant cylindriform silk

Juanita Francis[a]

[a] *Division of Pure and Applied Biochemistry, Lund University, Sweden*

Juanita.francis@tbiokemi.lth.se

Silk is a sustainable, versatile material and a suitable alternative to currently less efficient processed polymer materials, i.e., high density polyethylene. Despite the many uses of silk, spanning textile and biomedical industries, there is still a limited understanding of how this chemical and structural complex material is formed, consequently limiting its societal benefits potential. Specifically, the lack of knowledge in the fibrillation process of the cylindriform silk (α → β), a rapidly produced silk (which forms a spider's egg casing) compared to the well-studied dragline silk.

We aim to explore cylindriform silk proteins' rapid fibrillation process, the transient molecules involved, and the conditions that lead to α → β transition and develop a technology that controls protein α → β transitions, storage and stability. A deuterated recombinant cylindriform silk protein produced by employing green chemistry techniques will be used to explore the fibrillation process. This recombinant system allows for more control over the silk protein production and fibrillation compared to native silk. Subjecting the recombinant silk protein to a unique progressive acidification technique, a multimodal analysis tool called NUrF (**N**eutron small-angle scattering (SANS), **U**V-vis, **R**aman and **F**luorescence) can resolve intermediate structures, and one can extract rate laws throughout silk fibrillation **[1]**. SANS is an advantageous technique for long kinetic fibrillation studies as it does not destroy the silk sample, i.e., X-rays. SANS will provide mesoscale insights into silk proteins' size, shape, internal organisation, complex formation, and structural alignment during fibrillogenesis. Combined with the chemical information from UV and Fluorescence, our approach will give a unique understanding of the necessary steps needed to form spider silk for future sustainable polymer processing and storage.

References

[1] Dicko, C., et al., *NUrF – optimisation of multiple in situ simultaneous, autonomous characterization techniques with small-angle neutron scattering instrumentation.* Review of Scientific Instruments, 2020. **9**(7): p. 075111.

Abstract: 085

Magnetoelectric coupling of rare-earth orthotantalates

X. Zhang[a]*, N.D. Kelly*[a]*, C. Liua, S.E Dutton*[a]*, S.S. Saxena*[a]

[a]*Cavendish Laboratory, University of Cambridge, Cambridge, United Kingdom*

xz386@cam.ac.uk

Quantum multiferroic materials form a new and emerging area of physics where one expects to find emergence of novel quantum phases induced by subtle coupling between spin and charge degrees of freedom at low temperatures [1-2]. Experimental study of such phenomena is limited by the lack of model materials where magnetism and dielectric properties can be tuned using magnetic fields at low temperatures.

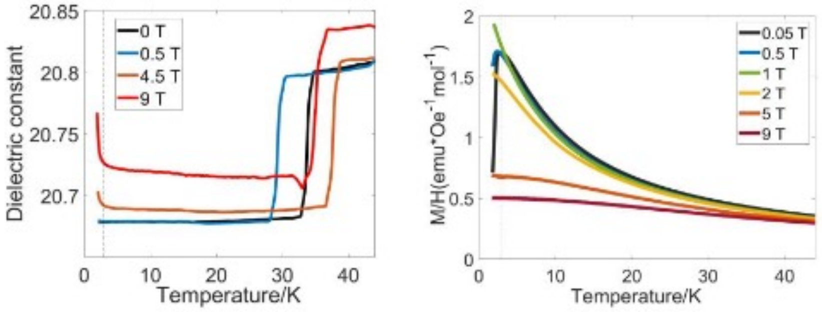


Fig. 1a: Increase of ε_r with applied field below the magnetic transition temperature of 2.25 K. **Fig. 1b**: Susceptibility curves in applied field. Transformation from AFM to FM with increasing field.

In a recent breakthrough, we found that TbTaO4 exhibits enhancement in dielectric response be- low 2 K on application of magnetic field, indicating magnetoelectric coupling. Previously, using susceptibility and heat capacity measurements we showed that TbTaO4 orders at TN = 2.25 K; powder neutron diffraction (PND) was used to solve the magnetic structure, which is A-type anti- ferromagnetic [3].

Polycrystalline M-TbTaO4 was synthesised by a solid-state reaction at 1500 0C. The zero-field magnetic heat capacity showed a sharp λ-type transition, typical of three-dimensional antiferromagnetic long-range ordering, at TN = 2.25 K, [3]. Variable-temperature PND was then carried out on the D1B and D2B beamlines at the ILL, Grenoble. Rietveld refinement revealed a commensurate k = 0 magnetic structure with Tb3+ spins parallel to the a-axis, Fig. 2. The onset of ordering is consistent with the heat capacity data and al- most the full Tb moment of 9 μB/formula unit is recovered by 1.5 K [3].

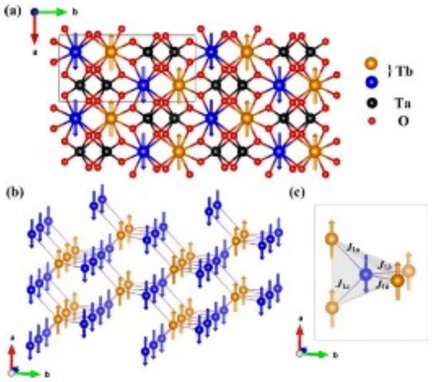


Fig. 2: Magnetic structure of TbTaO₄ showing the antiferromagnetic nearest-neighbour interactions [3].

References

[1] C. Morice et al., Physical Review B 96(24), 245104 (2017).

[2] A. Narayan et al., Nature Materials 18(3), 223-228 (2019). [3] N. D. Kelly et al., Physical Review Materials 6, 044410 (2022).

Towards a comprehensive picture of temperature-responsive elastin-like peptides

Olga Matsarskaia[a], Tatiana Morozova[a], Zeina Wehbe[a], Sarah Waldie[b], Nicolas A. Garcia[c], Michael M. Koza[a], Martine Moulin[a], Valerie Laux[a], Michael Haertlein[a], Trevor Forsyth[a], Jean-Louis Barrat[a], Felix Roosen-Runge[b]

[a]ILL, Grenoble, France
[b]University of Malmö, Sweden
[c]Universidad Nacional Del Sur, Argentina

felix.roosen-runge@mau.se

Elastin-like peptides (ELPs) are biomolecules mimicking the hydrophobic repeat units of elastin, a protein providing elasticity to biological tissues such as lung, ligaments and blood vessels. ELPs undergo a hydrophobic collapse upon crossing a lower critical solution temperature (LCST). Due to their stimulus-responsive properties, ELPs are of interest for a broad range of applications including advanced biomaterials, protein purification and drug delivery. While the hydrophobic collapse is believed to be key for the elastic properties of elastin, a comprehensive mechanistic characterisation of the static and dynamic aspects of the collapse has not yet been obtained. In particular, the dynamical state within the collapsed hydrophobic domains of elastin is debated (fluid-like structure vs. a more specific stacking). By combining SANS, QENS, molecular dynamics simulations and selective deuteration, we investigate the temperature response of selectively deuterated ELPs. Neutron data indicate differences in the behaviour of short and long ELPs, and, in agreement with simulations, a shift towards more compact ELP structures with increasing temperature is observed. Using our results, we aim at establishing a framework for the investigation of stimulus-responsive molecules and materials.

Abstract: 089

Facilities for macromolecular crystallography at the HZB

Thomas Hauß[a], T. Barthel[a], L. Benz[a], T. Crosskey[a], R. Förster[a], P. Fröling[a], C. Gless[a], M. Hellmig[a], F. Lennartz[a], U. Mueller[a], G. Weber[a], J. Wollenhaupt[a] & M. S. Weiss[a]

[a]Macromolecular Crystallography, Helmholtz-Zentrum Berlin für Materialien und Energie,
Albert- Einstein-Str. 15, D-12489 Berlin, Germany

hauss@helmholtz-berlin.

The Macromolecular Crystallography (MX) group at the Helmholtz-Zentrum Berlin (HZB) has been in operation since 2003. Since then, three state-of-the-art synchrotron beam lines (BL14.1-3) for MX have been built up on a 7T-wavelength shifter X-ray source [1-3]. Currently, the three beam lines represent the most productive MX-stations in Germany, with more than 3900 PDB depositions (Status 06/2022). BLs14.1 and 14.2 are energy tuneable in the range 5.5-15.5 keV, while beam line 14.3 is a fixed-energy side station operated at 13.8 keV. All three beam lines are equipped with state-of-the-art detectors: BL14.1 with a PILATUS3S 6M detector, BL14.2 with a PILATUS3S 2M and BL14.3 with a PILATUS 6M detector. BL14.1 and BL14.2 are in regular user operation providing close to 200 beam days per year and about 600 user shifts to approximately 100 research groups across Europe. Recently remote beamline operation has been established successfully at BL14.1 and BL14.2. BL14.3 has been equipped with a MD2 micro-diffractometer, a HC1 crystal dehydration device and a REX nozzle changer making it suitable for room temperature experiments. Additional user facilities include office space adjacent to the beam lines, a sample preparation laboratory, a biology laboratory (safety level 1) and high-end computing resources. Within this presentation/poster a summary on the experimental possibilities of the beam lines and the ancillary equipment provided to the user community will be given.

References

[1] U. Heinemann, K. Büssow, U. Mueller & P. Umbach (2003). Acc. Chem. Res. 36, 157-163.

[2] U. Mueller, N. Darowski, M. R. Fuchs, R. Förster, M. Hellmig, K. S. Paithankar, S. Pühringer, M. Steffien, G. Zocher & M. S. Weiss (2012). J. Synchr. Rad. 19, 442-449.

[3] U. Mueller, R. Förster, M. Hellmig, F. U. Huschmann, A. Kastner, P. Malecki, S. Pühringer, M. Röwer, K. Sparta, M. Steffien, M. Ühlein & M. S. Weiss (2015). Eur. Phys. J. Plus 130, 141-150.

Abstract: 091

MARMOT – Multiplexing on Thales

Paul Steffens[a], *Martin Boehm*[a], *Pierre Courtois*[a]

[a]*Institut Laue-Langevin, Grenoble*

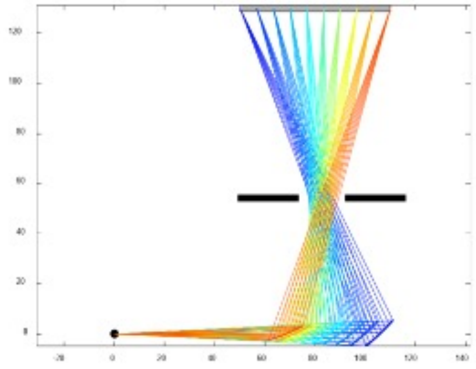
steffens@ill.eu

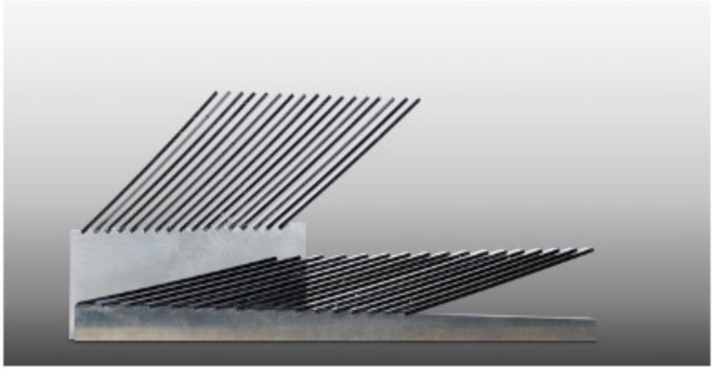
MARMOT (Multiplexed Array for Mapping on Thales) is a multiplexed secondary spectrometer designed for the cold triple-axis spectrometer Thales at the ILL.

As its predecessor FlatCone, it is foreseen as an interchangeable analyzer-detector unit replacing the classical secondary spectrometer of Thales for applications necessitating a coverage of about 1 to 2 Brillouin zones with simultaneous data collection in a range of final energies from about 3 to 6 meV. In practice, the data acquisition rate is expected to increase by about a factor 30 with respect to the standard unit.

It will use a set of bent Silicon blades as analyzer material. They are curved such that neutrons are scattered in an energy-dispersive way on a position sensitive analyzer. By aligning a high number of blades in a carefully designed geometry, neutrons coming from the sample are focused to one point on the detector, depending on their energy. This condition holds for a whole range of wavelengths and thus provides a truly continuous measurement of the neutron energy. This allows, in every channel, to measure the energy spectrum of the scattered beam at once.

Test measurements on a prototype confirm that the concept works as expected and with a very good energy resolution.





Solution structure of FRP-$^{\Delta NTE}$OCP^{O} complex in cyanobacteria

Mina Hajizadeh[a], Maksym Golub[a], Marcus Moldenhauer[b], Olga Matsarskaia[c], Anne Martel[c], Lionel Porcar[c], Dmytro Soloviov[d,e,f], Alexander Kuklin[d,e], Eugene G. Maksimov[g], Thomas Friedrich[b] and Jörg Pieper[a]

[a] Institute of Physics, University of Tartu, Tartu, Estonia,
[b] Technical University of Berlin, Institute of Chemistry, Berlin, Germany,
[c] Institut Laue-Langevin, Grenoble, France
[d] Joint Institute for Nuclear Research, Dubna, Russia
[e]Moscow Institute of Physics and Technology, Russia
[f] Institute for Safety Problems of Nuclear Power Plants NAS of Ukraine, Kyiv, Ukraine,
[g] M. V. Lomonosov Moscow State University, Department of Biophysics, Moscow, Russia

Mina.hajizadeh@ut.ee

The Orange Carotenoid Protein (OCP) is a photoactive protein that plays an essential role in high light tolerance in cyanobacteria. OCP is in the orange form in its ground state OCP^{o}. After photo-excitation, the carotenoid inside the OCP moves to its N-terminal domain, and the protein expands, resulting in the red active state OCP^{r}. The back conversion of OCP to the ground state OCP^{o} is facilitated by complex formation with the Fluorescence Recovery Protein (FRP). However, till today, no high-resolution tertiary structure of the OCP-FRP complex has been reported.

In the present study, we use a specific orange OCP mutant $^{\Delta NTE}$OCP^{orange} lacking the N-terminal extension and retaining the compact ground state structure, which can still bind FRP. This enables us to investigate the solution structure of the complex of $^{\Delta NTE}$OCP^{orange} with wild-type FRP utilizing small-angle neutron scattering coupled with size exclusion chromatography.

The data analysis proved that there are two different populations of the FRP-$^{\Delta NTE}$OCP^{orange} complex in the sample solution according to D_{max} -values (the maximum distance in the particle). One of the populations has a more extended structure than the other. We used Sasview and Pepsi software to find the best structures for these populations in the next step. In the population with longer D_{max}, we have a 2:2 complex between $^{\Delta NTE}$OCP^{orange} and FRP, while the other population is a 2:1 complex. A tertiary structure has been proposed for each population.

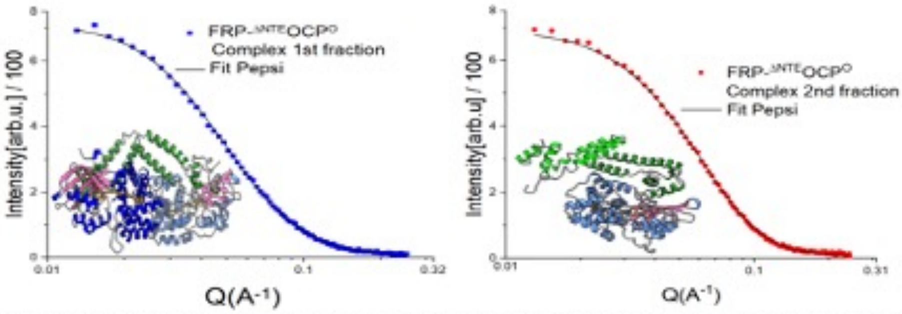


Figure 1. SANS data of FRP-$^{\Delta NTE}$OCP^{O} complex at a protein concentration of about 1 mg/ml after size exclusion chromatography; the Blue squares are SANS data of the first fraction of the SEC experiment, and the red circles are the second fraction of the SEC experiment, measured at room temperature using the instrument ILL_d22. Black solid lines represent the fitting curves of the PepsiFlex model for the first and the second fraction. Pepsi modeling was carried out using the crystal structure of OCP^{O} (PDB: 3MG1) with removed NTE and FRP dimer (PDB: 4JDX).

Abstract: 093

Extending MIEZE spectroscopy towards thermal wavelengths

Johanna K. Jochum[a, b], Christian Franz[c], Christian Pfleiderer[b,d]

[a]Heinz Maier-Leibnitz Zentrum, Technische Universität München, D-85748 Garching, Germany
[b]Physik Department, Technische Universität München, D-85748 Garching, Germany
[c]Jülich Centre for Neutron Science JCNS-MLZ, Forschungszentrum Jülich GmbH Outstation at MLZ
FRM-II, D-85747 Garching, Germany
[d]Zentrum für QuantumEngineering (ZQE), Technische Universität München, D-85748 Garching, Germany

jjochum@frm2.tum.de

We propose a MIEZE (Modulation of intensity with zero effort) set-up for high-resolution neutron spectroscopy at momentum transfers up to 3Å-1 and energy transfers up to ~20meV using thermal neutrons. Profiting from the general advantages of the MIEZE technique, notably a lack of sensitivity to spin depolarizing samples and sample environments as well as strongly incoherent scattering, the use of thermal neutrons increases the range of validity of the spin-echo approximation towards shorter spin-echo times. In turn, the thermal MIEZE option for greater ranges (TIGER) closes the gap between classical neutron spin-echo spectroscopy and conventional high- resolution neutron spectroscopy techniques such as triple-axis, time-of-flight, and back-scattering. To illustrate the feasibility of TIGER we present the details of an implementation at the beam- line RESEDA at FRM II by means of an additional velocity selector, polarizer and analyzer.

Abstract: 094

Structural study of the Hepatitis B virus X protein and its interactome

Alexis Clavier[a]

Technische Universität München, D-85748 Garching, Germany

alexis.clavier@tum.de

My project focuses on the Hepatitis B virus (HBV) which is responsible for up to 300 million cases per year and hundreds of millions of chronic infections[1]. Moreover, the WHO estimated in 2019 that the virus resulted in 820,000 deaths[1]. This high level of mortality of the virus is mainly linked to liver tissue damage associated with the chronic infection[1].

The Hepatitis B virus belongs to the *Hepadnaviradae* family of viruses[2]. The HBV genome contains only 3.2 kilobases and encodes four overlapping open reading frames encoding: the envelope proteins, the capsid-forming core proteins, the viral polymerase, and the HBx protein[2]. The X ORF, the smallest of the genome, encodes for the Hbx protein of 154 amino acids (17 kDa), which is presumed to be at least partially intrinsically disordered[2]. The disordered propensity of HBx engendered vain attempts to crystallize the protein except for shorts helical peptides[5,6]. Even today, only short helical structures in the amino and carboxyl-terminal are described[7]. Despite its short size, the HBx has been reported to interact with a hundred of different partners[9]. The flexibility of HBx has been linked to its functional breadth from transactivation to the regulation of apoptotic signals[10]. The molecular mechanisms underlying the ability of HBx to engage a wide range of host cell proteins remain uncharacterized in the absence of experimental setups and *in vitro* methods to investigate HBx protein structurally[2]. The multifunctionality of HBx also plays a role in the pathogenesis of viral-induced hepatocellular carcinoma (HCC) – one of the leading causes of death induced by cancer in the modern world[11].

My project aims to study, thanks to NMR combined with biophysical assays, the structural propensities of HBx protein in isolation and in complex with some of the interaction partners.

1. We would like to confirm our model with the complex HBx-DNA damage-binding protein 1 (DDB1). The binding HBx-DDB1 is hypothesized to be a mandatory condition for the replication of the virus[12]. It enables HBx to redirect the immunity complex SMC5/6, which binds to the viral DNA to block the replication, to the proteasome for degradation[12].

2. We would like to transfer these methods to further describe the interaction between HBx and three host cell protein reported to be HBx targets: - the protein HSP60 from the heat shock family[13], - the Bcl-XL anti-apoptotic protein[6], - the tumor suppressor P53[14]. These three proteins are indirectly or directly linked to hepatocellular carcinoma by regulating the cell cycle of the apoptosis pathway[11].

3. The existence of a truncated isoform of HBx and of multiple mutations inside the hepatocellular carcinoma liver cells in the context of chronic infection have been very well documented[15,16]. These modifications of the HBx sequence are highly interesting for a better understanding of how the structure of the protein impacts the different pathways but also to imagine new drugs in order to counter the HCC progression[16].

References

1. https://www.who.int/news-room/fact-sheets/detail/hepatitis-b.
2. Song, H *et al. Cell & Mol. Imm*, **18**, 1345-1346 (2021).
3. Ali, A. *et al.* World J. Gastroenterol, **20**, 10238-10248 (2014)
4. Suh, A. *et al. PLoS Genet.* **10**, e1004559 (2014).
5. Li, T. *et al. Nat. Struct. Mol. Biol.* **17**, 105–111 (2010).
6. Zhang, T.-Y. *et al. Nat. Commun.* 1–14 (2019).
7.Hernández, S. *et al. Biomedicines* **9**, 1701 (2021).
8.Ramakrishnan, D. *et al. J. Virol.* (2019).
9.Zhang, T. *et al. J. Proteomics* **84**, 92–105 (2013).
10. Schuster, R. *et al. Oncogene* **21**, 6606–6613 (2002).
11. Sivasudhan, E. *et al. Cells* **11**, 741 (2022).
12.Kim, W., *et al. J. Virol.* **90**, 9644–9653.
13.Zhang, S. M. *et al Arch. Virol.* **150**, 1579–1590 (2005).
14.Elmore, L. W. *et al. Proc. Natl. Acad. Sci. U. S. A.* **94**, 14707– 14712 (1997).
15. Kumar, V., *et al. Proc. Natl. Acad. Sci. U. S. A.* **93**, 5647–5652 (1996).
16. Ma, N.-F. *et al. Clin. Cancer Res. Off. J. Am. Assoc. Cancer Res.* **14**, 5061–5068 (2008).

Abstract: 100

Polarized neutron diffraction to probe local magnetic anisotropy and magnetization density

I. Kibalin[a], *A. Gukasov*[a]

[a]*Laboratoire Léon Brillouin, CEA-CNRS, CE-Saclay, 91191 Gif-sur-Yvette, France*

iurii.kibalin@cea.fr

Polarized neutron diffraction (PND) has proven to be one of the most efficient tools to enlighten the magnetic interaction mechanisms in molecule-based materials. In the past decades, PND was largely used to study spin density distribution in paramagnetic molecular crystals [1]. Many efforts have been devoted to the analysis of PND data in highly anisotropic paramagnets. It is becoming a reference in mapping the magnetic anisotropy at the atomic scale in molecular magnets [2].

In spite of the obvious success in probing the magnetic anisotropy by PND, it should be noted that the used refinement procedure employs the so-called dipole approximation for the magnetic form factor. It assumes that the spatial distribution of the magnetization density of an ion is isotropic, which is valid as long as it is larger than approximately the mean radius ⟨r⟩ of the unpaired electrons. Recently the magnetization density has been reconstructed from PND data for a single crystal of $Yb_2Ti_2O_7$ pyrochlore by maximum entropy method (MEM). The strong asphericity of magnetization density has been clearly observed for Yb^{3+} ion (Fig. 1). The radial distributions reconstructed by MEM reproduce rather well the theoretical radial distribution of Yb^{3+} ion. It was found, that the shapes of both magnetic distributions for Yb^{3+} ion are in qualitative agreement. The reconstructed distribution can now be confronted with the theory predictions.

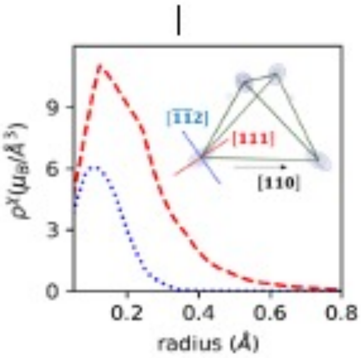


Fig. 1: The magnetization density of Yb^{3+} along [111] axis (red dashed lines) and along $[\bar{1}\bar{1}2]$ axis (blue dotted lines) for Yb_2Ti_2O7 compound at 5 K in 1 T after maximum entropy reconstruction. The density iso-surfaces of four Yb^{3+}-ions are shown in insets.

References

[1] E.Ressouche et al, JACS 115, 3610 (1993); V. Baron et al, JACS 119, 3500 (1997); J.A. Stride et al, C. R. Acad. Sci. Paris, Chimie / Chemistry 4, 105–112 (2001) ; B.Gillon, Inorganic Chemistry 46, 1090 (2007).

[2] K.Ridier, M. Abhishake et al., Angewandte Chemie International Edition, 2016, 55, 3963–67.

[3] I.A. Kibalin and A. Gukasov, Physical Review B, 2022, 105, 104411.

Protein short-time diffusion in polydisperse crowding

Tilo Seydel[a], Christian Beck[a,b], Felix Roosen-Runge[c], Hender Lopez[d], Marco Grimaldo[a], Martin Oettel[b], Jean-Louis Barrat[e], Frank Schreiber[b]

[a]Institut Max von Laue-Paul Langevin, Grenoble, France
[b]University Tübingen, Tübingen, Germany,
[c]University Malmö, Malmö, Sweden
[d]Technical University of Dublin, Ireland
[e]University of Grenoble-Alpes, France

seydel@ill.eu

Knowledge of the protein tracer diffusion constitutes a key element to describe intracellular transport, which can be modeled by the self-diffusion in colloid systems [1]. However, it is necessary to test the underlying assumption that neither the protein shape and size nor the polydisperse nature of the cytosol matter.

We present a combined experimental and simulation study of the protein tracer diffusion in deuterated *Escherichia coli* cellular lysate [2] (figure 1). Quasi-elastic neutron scattering accesses the short-time diffusion of immunoglubulin (IgG) in this lysate. Varying the mixing ratio and volume fraction of IgG and lysate, we observe that this diffusion only depends on the total volume fraction of macromolecules. Stokesian dynamics simulations confirm that when the tracer size agrees with the average size of the polydisperse lysate, these proteins are indeed slowed down similar to a monodisperse solution of same volume fraction. In contrast, larger/smaller proteins diffuse slower/faster, respectively. IgG being close to this average size, we obtain a consistent picture on the diffusion from simulations and experiments.

Ongoing investigations with different tracer proteins in lysate as well as binary mixtures of proteins support this colloid picture of the self-diffusion even in such complex polydisperse cell-like environments, which is promising for a future quantitative understanding of reaction pathways in biology.

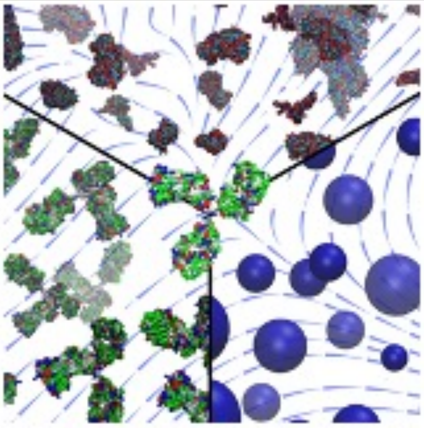


Figure 1: Schematic of an IgG tracer protein in different actual and modeled crowding settings [2], namely self-crowding by varying the concentration of the concentration itself (bottom left), or polydisperse crowding by different macromolecules (top), modeled by poydisperse hard spheres (bottom right).

References

[1] M. Grimaldo et al., Quart. Rev. Biophys. **52** (2019) e7, 1

[2] M. Grimaldo et al., J. Phys. Chem. Lett. **10** (2019) 1709

Polyelectrolyte/Surfactant Complexes (PESCs) for controlling rheological behaviour – relation between mesoscopic structure and macroscopic rheology

Michael Gradzielski[a], Olga Kuzminskaya[a], Ingo Hoffmann[b]

[a]Stranski Laboratorium für Physikalische Chemie, Technische Universität Berlin, Germany
[b]Institute Laue-Langevin, Grenoble, France

michael.gradzielski@tu-berlin.de

Mixtures of oppositely charges polyelectrolyte and surfactant in general form polyelectrolyte/ surfactant complexes (PESCs), whose structures may vary largely depending on the detailed molecular structure of the constituting polyelectrolyte and surfactant, as well as on the strength of interaction. For instance, it has been observed that addition of conventional anionic surfactants like SDS or LAS to cationically modified cellulose (JR400) leads to a marked increase of viscosity by 3- 4 orders of magnitude, while for the case of PDADMAC only a very tiny viscosity increase is noticed. Investigation by SANS experiments employing the technique of contrast variation (using deuterated surfactant) shows a very different mesoscopic structure to be present for the case of JR400 and PDADMAC. For JR400 intermixed rodlike aggregates are formed, while PDAMDAC is just decorating similarly structured micelles (see Fig. 1). This largely different aggregation behaviour was then also studied with respect to its dynamical properties by neutron-spin echo (NSE) experiments that showed also a substantially different dynamic behaviour. In summary, the very different macroscopic appearance of the samples can be explained well by their mesoscopic structure that depends largely on the detailed structure and properties of the polyelectrolyte employed. As such formulations are highly relevant for many application, this has to be considered in thorough detail when designing such formulations.

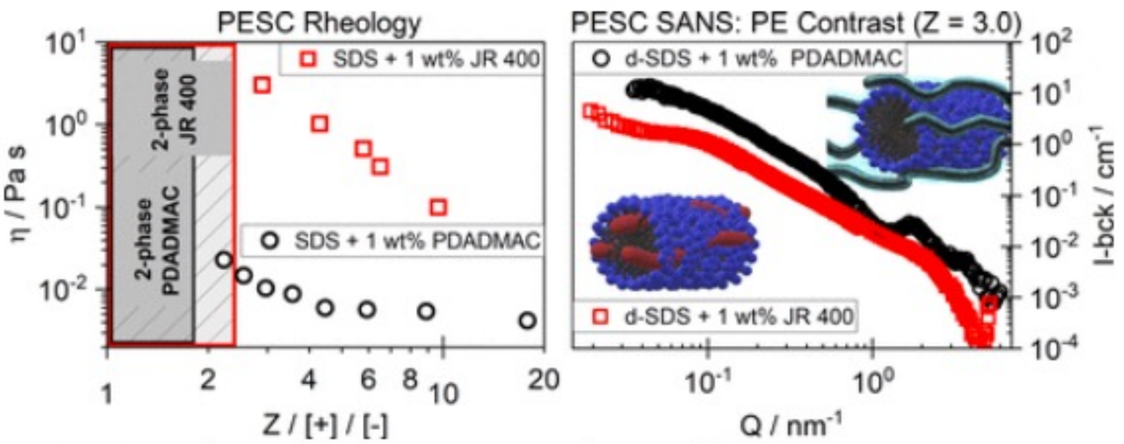


Fig.1: Viscosity and SANS curves in PE contrast for complexes with JR400 or PDADMAC. [1]

References

[1] O. Kuzminskaya, I. Hoffmann, D. Clemens, M. Gradzielski, Macromolecules 2021, 54, 4615

Surgical control of quantum matter: uniaxial pressure for neutrons

G. Simutis[a,b], Y. Sassa[b], A. Bollhalder[a], M. Zolliker[a], J. Küspert[c], D. Bucher[c], M. Bertelsen[d], M. Månsson[e], M. Boehm[f], F. Bourdarot[f], J. Chang[c], N. B, Christensen[g], M. Bartkowiak[a], D. Mazzone[h], M. Janoschek[a]

[a]Paul Scherrer Institut (PSI), Laboratory for Neutron and Muon Instrumentation, CH-5232 Villigen PSI, Switzerland,
[b]Department of Physics, Chalmers University of Technology, SE-41296 Göteborg, Sweden,
[c]University of Zürich, Laboratory for Quantum Matter Research, CH-8057 Zürich, Switzerland
[d]European Spallation Source ERIC, P.O. Box 176, SE-221 00, Lund, Sweden
[e]Department of Applied Physics, KTH Royal Institute of Technology, SE-106 91 Stockholm, Sweden
[f]Institut Laue-Langevin, 71 avenue des Martyrs, CS 20156, 38042 Grenoble Cedex 9, France
[g]Department of Physics, Technical University of Denmark, DK-2800 Kongens Lyngby, Denmark
[h] Paul Scherrer Institut (PSI), Laboratory for Neutron Scattering, CH-5232 Villigen PSI, Switzerland

yasmine.sassa@chalmers.se

Uniaxial pressure is a particularly clean parameter for tuning quantum matter. By explicitly breaking the symmetry of the system and selectively tuning relevant interactions, it allows discovery, understanding and control of novel phases in materials with strong correlations.

In our recent experiments, we have shown that the uniaxial pressure can be used efficiently with neutron scattering techniques. Combining novel neutron instrumentation with simple uniaxial pressure cells enabled us to unequivocally show that the underlying ground state in the cuprate superconductors is generated by the uniaxial charge-spin stripes and that we can selectively populate different domains of the electronic order [1].

To pursue the systematic application of the uniaxial pressure for neutron experiments we are developing in-situ devices, which will be suitable for a wide range of scattering experiments in the current neutron facilities but also geared towards use in the superior source of the ESS. In this contribution, we will present a recently commissioned uniaxial cell that is optimized for small- angle experiments (hard X-ray and SANS) [2] and our plans for the universal uniaxial pressure device.

References

[1] G. Simutis et al., in review and arXiv:2204.02304 (2022)
[2] G. Simutis et al., in preparation

High performing computing infrastructure and use cases for neutron scattering research

Johan Hellsvik[a], *Henric Zazzi*[a], *Dirk Pleiter*[a]

[a]*PDC Center for High Performance Computing, KTH Royal Institute of Technology, SE-106 91 Stockholm, Sweden*

hellsvik@kth.se

High Performance Computing (HPC) capabilities are becoming increasingly important in the context of large-scale experimental facilities like ESS. They can be used for compute-intensive analysis of the data produced by such facilities. Still even more important is the use of HPC to address the challenge to relate experimental data and theory, e.g. in the area of condensed matter physics. Comparison between experimental results and atomistic models is not straightforward and nuanced by both experimental effects and theoretical approximations. This is an especially challenging issue for understanding spectroscopic data.

In this contribution we highlight how the infrastructure and services provided by the PDC Center for High Performance Computing can contribute to research performed at neutron scattering facilities, with examples of use cases for energy materials, magnetic materials, and superconductivity.

The HPE Cray EX supercomputer **Dardel** [1] is a flagship system operated at PDC for academic and business users. **Dardel HPC CPU partition** *(in operation)* offers the community a high performance computing platform for science and engineering. Dardel features a large software stack for modelling condensed matter physics [2]. **Dardel HPC GPU partition** *(starting autumn 2022)* will significantly extend the overall capabilities of Dardel and provide a powerful resource, e.g., for processing experimental data by means of machine learning algorithms. **Dardel Cloud** based OpenStack based private cloud will allow users to deploy workflows which are not meant to run on traditional HPC systems, for instance less compute-intensive workflows, interactive portals, and database servers and frameworks (*starting autumn 2022*).

In collaboration with research groups at Swedish universities, Nordita, and the ESS Data Management and Software Center, PDC is striving to contribute to a data driven e-infrastructure that will combine comparison of experimental data S($\mathbf{q}$) and S($\mathbf{q}$,ω) with state-of the-art atomistic simulation and materials informatics [3,4], and will reduce the barrier to entry for direct comparison of simulation data with data from experiments.

References

[1] Dardel https://www.pdc.kth.se/hpc-services/computing-systems
[2] Materials Theory Codes on Dardel at PDC, J. Hellsvik, O. Eriksson,and I. Di Marco, https://www.pdc.kth.se/publications/pdc-newsletter-articles/2021-no-2
[3] Spin wave excitations of magnetic metalorganic materials. J. Hellsvik, R. Díaz Pérez, R. M. Geilhufe, M. Månsson, A. V. Balatsky, Phys. Rev. Materials 4, 024409 (2020)
[4] Organic Materials Database (OMDB) https://omdb.mathub.io/

Charge transfer in a redox protein – insights from neutron dynamics studies and cryo-neutron crystallography

Juliette M. Devos[a], *Maxime G. Cuypers* [b,c], *Estelle Mossou*[c,d], *Marie-Hélène Lemée*[e], *Tilo Seydel*[f], *Edward P. Mitchell*[d], *Michael Härtlein*[a], *V. Trevor Forsyth*[a,c,g]

[a]*Life Sciences Group, Institut Laue-Langevin, Grenoble, France*
[b]*St. Jude Children's Research Hospital, Memphis, TN, USA*
[c]*Faculty of Natural Sciences, Keele University, Staffordshire ST5 5BE, UK*
[d]*European Synchrotron Radiation Facility, Grenoble 38043, France*
[e]*Diffraction Group, Institut Laue-Langevin, Grenoble, France*
[f]*Spectroscopy Group, Institut Laue-Langevin, Grenoble, France*
[g]*Faculty of Medicine and LINXS Institute for Advanced Neutron and X-ray Science, Lund, Sweden*

devosj@ill.fr

Rubredoxins are small monomeric iron-sulphur cluster proteins found in prokaryotes and in some eukaryotes. They act as electron carriers in a variety of biological activities such as fatty acid metabolism, processing of reactive oxygen species, and carbon fixation. Rubredoxin from *Pyrococcus furiosus* (Pf) is one of the most thermostable proteins known. As a redox protein, rubredoxin is also an important model system for an understanding of electron transfer processes associated with catalysis.

In previous work [1], a near atomic resolution study of perdeuterated Pf rubredoxin was carried out. This study recorded several hydronium ions that are clearly implicated in the stability of both redox forms of the protein. We now present recent and unpublished studies on this exciting system. Using neutron spectroscopy, we have measured both inelastic and elastic fixed window data in order to investigate the possibility of tunnelling phenomena within the protein. While the data needs further careful analysis, the dynamical transition of the protein has been clearly identified. We also have recorded two high resolution (0.88 Å and higher) single crystal neutron diffraction experiments of perdeuterated rubredoxin at a temperature of 100 K. The presented work was carried out using the high resolution thermal diffractometer D19 at the Institut Laue-Langevin, with a wavelength of 1.49 Å for the first data set and a wavelength of 1.17 Å for the second one. The protein was produced in the Deuteration Laboratory of ILL's Life Sciences Group [2]. The crystal growth, the sample mount and the tests carried out in order to obtain the best possible cryo data set will be highlighted. The structural analysis shows a striking network of Zundel and hydronium ions as well as ordered water molecules that link to the iron-sulphur cluster of the protein. These observations are highly suggestive of the way in which protonation shifts involving exotic ionic species may be involved in the charge transfer processes of redox proteins. The results are the first of their type in the study of redox biology.

References

[1] M.G. Cuypers; S.A. Mason; M.P. Blakeley; E.P. Mitchell; M. Haertlein; V.T. Forsyth, *Angew. Chem.* **2013**, *52*, 1022-1025.
[2] M. Haertlein; M. Moulin; J.M. Devos; V. Laux; O. Dunne; V.T. Forsyth, *Methods Enzymol.* **2016**, *566*, 113-157.

Abstract: 109

The ILL Deuteration Laboratory (ILL D-Lab)

Valérie Laux[a], Martine Moulin[a], Juliette M. Devos[a], Michael Härtlein[a], V. Trevor Forsyth[a,b]

[a]Life Sciences Group, Institut Laue-Langevin, Grenoble, France
[b]Faculty of Medicine and LINXS Institute for Advanced Neutron and X-ray Science, Lund, Sweden

dlab-proposals@ill.fr

The ILL Deuteration Laboratory is a platform dedicated to isotope labelling of biological molecules. More specifically, the D-Lab team members are experts in the deuteration of biomolecules for neutron applications, such as neutron scattering, protein crystallography, dynamics and reflectometry. The D-Lab is part of the ILL Life Sciences Group within the Partnership for Structural Biology (PSB) located in Grenoble, France. It is run as a user platform available to all neutron users. Access to the platform is by a rapid electronic peer-review system, available at any time.

In neutron experiments in biology, the replacement of the common hydrogen isotope protium (^1H) by its stable isotope deuterium (^2H) is of crucial importance for biomolecules [1]. Depending on the neutron experiment, various levels of deuteration of these molecules are necessary [2]. Microorganisms such as bacteria and yeasts have been successfully adapted to growth in deuterated minimal media. Large-scale protein deuteration by recombinant expression in high-cell density cultures was initially developed in the ILL D-Lab. The production of various labelled biomolecules required for the study of proteins, protein-nucleic acid complexes, protein-lipid complexes, glycoproteins, membrane proteins and stealth lipid nanodiscs will be presented. The *in vivo* deuteration of small biomolecules of major functional importance will also be highlighted, as well as recent advances and method developments for the deuteration of biomolecules *in vivo* and *in vitro*.

For further information, you can consult the webpage on the ILL website dedicated to the platform (https://www.ill.eu/users/support-labs-infrastructure/deuteration-laboratory). The ILL D-Lab team (dlab-proposals@ill.fr) can be contacted at any time and is fully available to assist neutron users in biology with their sample preparation.

References

[1] M. Haertlein; M. Moulin; J.M. Devos; V. Laux; O. Dunne; V.T. Forsyth, *Methods Enzymol.* **2016**, *566*, 113-157.
[2] O. Dunne; M. Weidenhaupt; P. Callow; A. Martel; M. Moulin; S. J. Perkins; M. Haertlein; V. T. Forsyth, *Eur. Biophys. J.* **2017**, *46*, 425–432.

Abstract: 110

Neutron scattering studies of organic cation dynamics in low-dimensional metal halide perovskites

Kanming Shi[a], Jiajun Qin[b], Weidong Cai[b], Feng Gao[b], Maths Karlsson[a]

[a]Dept. Chemistry and Chemical Engineering, Chalmers University of Technology, Sweden

[b]Dept. Physics, Chemistry and Biology, Linköping University, Sweden

kanming@chalmers.se

Metal halide perovskites (MHPs) are currently receiving considerable attention because of their great potential for use in optoelectronic technologies, such as environmental friendly solar cells and light emitting diodes. However, a low stability towards heat and moisture remain a big hindrance for using these materials in practical devices and, furthermore, the mechanism of their high photoelectric conversion efficiency remains unclear. While by far, the most studied MHPs are based on the three-dimensional perovskite structure, recent research efforts have been focused on lower dimensional systems. Crucially, these materials generally show better stability towards heat and moisture than their three-dimensional counterparts. Yet, a critical challenge in this field pertains to understanding the dynamics of the organic component in these types of materials. Here, we report on investigations of the organic cation dynamics in the low-dimensional chiral/achiral perovskites (R/S-MBA2MnBr4 and RAC-MBAMn2Br5EtOH, MBA+ is the methylbenzylamine C6H5CH(CH3)NH3+, EtOH is the ethanol C2H5OH), using quasi- elastic neutron scattering (QENS) and inelastic neutron scattering (INS). These perovskite materials which have new compositions and structures show different photoluminescence (PL) properties, and the similar situation occurs between the perovskites which have the same composition but chiral symmetrical cations. We believe these unexplored phenomena which can affect the optoelectronic properties are related to their cation dynamics. By monitoring the intensity of the quasielastically scattered neutrons upon heating the sample from 44 K to 375 K, we can find different kinds of cation motions including different kinds of 3-site rotation jump diffusions (the hydrogens from the same methyl or amine have 3 equivalent sites, and they can move to next equivalent sites under conditions such as appropriate temperature). With the temperature increasing, we can find the elastic incoherent structure factor is decreased and more kinds of localized motion including phenyl flip and whole cation tilt could be found. These motions might be the reason which leads the PL intensity decreases and the emission peak is slightly blue shifted because of the potential energy transfer and band gap change.

Surfactant self assembly in halogen-free deep eutectic solvents

Elly K. Bathke[a], *Sylvain Prévost*[b], *Karen J. Edler*[c]

[a]*University of Bath, Bath, United Kingdom*
[b]*Institut Laue-Langevin, Grenoble, France*
[c]*Lund University, Lund, Sweden*

Ekbathke@gmail.com

Deep eutectic solvents (DES) are an emerging class of modern, often "green" solvents, that can be formed by mixing a different components, often quaternary ammonium salts with sugars, metal salts, carboxylic acids, amino acids or alcohols, to create hydrogen bonded room temperature liquids with a decreased melting point in comparison to their components. They can exhibit unique properties and high tailorability, making them interesting in a wide range of fields such as for electrodeposition, synthesis, extraction or for pharmaceutical applications. DES have been found to support the self assembly of a range of amphiphilic molecules, and the type of surfactants and interaction of the surfactant molecule with the solvent components, as well as additives, have been found to influence the size, shape and morphology of the self-assembled structures formed. Research is also being conducted into their application for e.g. templating of mesoporous materials or emulsification for transdermal drug delivery.

Most of the commonly used DES are formed from components that contain halide ions, and are often not compatible with applications such as catalysis, where the presence of halide ions can poison the catalyst. Here we present the formation and structures of cationic and, not previously in type III DES reported, non ionic surfactant micelles in glycerol based halogen free DES systems, investigated through small angle neutron scattering. Factors such as DES components and composition, and surfactant head group and tail length will be discussed.

On the synergy between neutron reflectometry and solid-state NMR

Ildefonso Marín Montesinos[a], Daniel Pereira[a], Andreas Santamaria[b], Marina Ilkaeva[a], João Pereira[a], Nathan Zaccai[c], Phillip Gutfreund[b], Mariana Sardo[a], Armando Maestro[d,e], Luís Mafra[a]

[a]CICECO-Aveiro Institute of Materials, University of Aveiro, Aveiro, Portugal
[b]Institut Laue-Lange- vin, Grenoble, France
[c]CIMR, University of Cambridge, Cambridge, United Kingdom
[d]Centro de Física de Materiales (CSIC, UPV/EHU)-Materials Physics Center MPC, San Sebastián, Spain
[e]IKER- BASQUE—Basque Foundation for Science, Plaza Euskadi 5, Bilbao, 48009 Spain

imarin@ua.pt

Nowadays, the successful structural determination of heterogeneous and complex molecular systems plays an important role in their understanding in many scientific areas as material science or structural biology. The combination of different characterization techniques has become compulsory to successfully study these complex systems. Having that in mind, our group has started to explore the complementary use of neutron scattering techniques and solid-state Nuclear Magnetic Resonance (ssNMR) for the characterization of heterogeneous molecular systems in two different contexts: i) porous materials for gas adsorption and ii) the interaction between lipid membranes and endocytic proteins from Clathrin-mediated endocytosis (CME).

Firstly, we show the use of neutron reflectometry (NR) to characterize the degree of undesired cross-linking reactions in the grafting reaction of propylamine in mesoporous silica SBA-15 using two different synthetic methods. The first methodology uses 3-aminopropyltriethoxysilane (APTES) in order to obtain an amine-functionalized SBA-15. The second synthetic strategy uses the same primary amine but with the amine group protected as a 3-triethoxysilylpropyl-tert-butylcarbamate (TESPtBc). After grafting of TESPtBc, the tert-butyl group is removed by acid treatment. The NR profiles clearly show the absence of cross-linking reaction when using TESPtBc as amination agent, while in the case of using APTES, cross-linking amine reactions seem to be dominant according to the NR profiles. These results are used to complement ssNMR studies of CO2 adsorption amine-functionalized silicas.

Secondly, the interaction between the adaptor protein CALM (clathrin assembly lymphoid myeloid leukemia) with the PiP2 recognition lipid is studied by NR and ssNMR. NR experiments are per- formed in lipid bilayers deposited on a solid substrate while ssNMR is performed in liposomes. Both experimental techniques provide complementary information of the binding of CALM to PiP2. The NR profiles show an interaction of the protein with the lipid hydrophilic heads with almost no penetration in the lipid bilayer. These results are supported by ssNMR measurements performed in liposomes containing PiP2. ssNMR spectra exhibit a strong chemical shift disturbance in the 13C and 1H resonances of the lipid head. Additionally, due to the atomic detail information given by NMR, nearby lipids surrounding PiP2 are also perturbed by the CALM interaction.

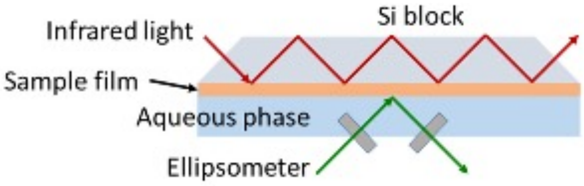
Implementation of simultaneous *in situ* ellipsometry and infrared spectroscopy at the ESS reflectometer beamlines

Thomas Ederth[a], *Béla Nagy*[a], *Daniel Merkel*[b]

[a]*Linköping University, Division of Biophysics and Bioengineering, IFM, Linköping, Sweden*
[b]*Wigner Research Centre for Physics, Budapest, Hungary*

thomas.ederth@liu.se

Neutron reflectivity (NR) has unique advantages for structural characterization of thin films and interfacial layers, providing thickness, composition, and roughness of composite layers or adsorbed films, on molecular length scales. However, the lack of chemical information, the limited time resolution, and difficulties in determining large thicknesses, are evident disadvantages, and many problems in soft matter science would benefit from a resolution of these limitations. For this purpose, we offer a sample environment for *in situ* optical characterization of solid-liquid interfaces at NR beamlines, integrating i) neutrons for structural analysis, ii) infrared spectroscopy for determination of chemical information and molecular conformation, and iii) spectroscopic ellipsometry (SE) for rapid and independent measurement of layer thicknesses and refractive in- dices. Combined NR/IR [1,2] and NR/ellipsometry [3] setups have been implemented in the past, and building on recent developments in liquid flow cells integrating NR/IR/SE [4] we have de- signed a modular setup for the ESS beamlines FREIA and ESTIA. To benefit from the expected rapid sample turnover at these beamlines, facilities for using the optical techniques in combination with sample changers are also explored. A prototype of the setup has been tested using a model system featuring pH-dependent swelling of polyelectrolyte layers. Potential benefits and limitations of the combined system will be discussed, including the uses of IR data, and the possibilities of co-fitting NR and SE data for improved structural characterization.



Schematic of the optical layout of the combined set-up, using a trapezoidal silicon block for infrared spectroscopy under attenuated total reflection (IR-ATR). The neutron beam traverses the block in the direction perpendicular to the paper.

References

[1] M. Strobl, R. Steitz, M. Kreuzer, M. Rose, H. Herrlich, F. Mezei, M. Grunze, R. Dahint, *Review of Scientific Instruments*, 2011, 82, 055101-055109.
[2] M. W. A. Skoda, B. Thomas, M. Hagreen, F. Sebastiani, C. Pfrang, *RSC Adv*, 2017, 7, 34208-34214.
[3] F. Sebastiani, R. A. Campbell, C. Pfrang, *RSC Adv*, 2015, 5, 107105-107111.
[4] C. Busch, B. Nagy, A. Stöcklin, P. Gutfreund, R. Dahint, T. Ederth, "A mobile set-up for simultaneous and in situ neutron reflectivity, infrared spectroscopy, and ellipsometry studies", *in manuscript*.

Abstract: 120

Nuclear structure studies at the FIPPS instrument at ILL

Giacomo Colombi[a]

[a]Institut Laue-Langevin, France

colombi@ill.fr

The FIssion Product Prompt gamma-ray spectrometer (FIPPS) is the new nuclear physics instrument at the *Institut Laue-Langevin* (ILL). FIPPS takes advantage of an intense pencil-like neutron beam (flux 108 n/s/cm2) for inducing neutron capture and neutron induced fission reactions and study the nuclear structure via high resolution gamma-ray spectroscopy. The array is composed by 8 Compton suppressed HPGe clover detectors. Ancillary devices are possible, as LaBr3 detectors for fast timing measurements or additional clover detectors (from the IFIN-HH collaboration) to increase efficiency and granularity.

After a general introduction on the main features of the instrument, recent developments to improve the energy resolution and the sensitivity of the instrument for fission studies will be reported. In particular, the procedure and effects of the correction for the cross-talk among the crystals in a same clover will be reported.

The setup and results from the first test of a diamond-base active target for neutron induced fission will be reported. These results will be shown with the ones from the well-established scintillator-based active target used at FIPPS in recent campaigns. The use of a fission tag allows for an identification of transitions from weak branches and/or isotopes produced with small fission yields.

Finally, the recently developed GEANT4 simulation code will be presented, with particular focus on the angular correlation analysis with hybrid gamma-ray arrays and on the first simulations for the development of a plunger device for lifetime measurements in fission fragments.

The GEANT4 simulations also allowed to analyze the scintillator-based active target data in order to extract lifetimes in the sub-ps timescale in fission fragments with the so-called *lineshape analysis*. This method will be presented as well as new results.

Chalcogenides for wearable thermoelectrics

Miloš Fejerčák[a], Michael Marek Koza[a]

Institut Laue-Langevin,71 avenue des Martyrs, CS 20156, 38042 GRENOBLE Cedex 9 - France

fejercak@ill.fr

Wearable renewable energy generators are an attractive alternative to battery-based systems and can generate power up to a few Watts for portable electronic equipment. Although the most inorganic semiconductor materials are brittle at room temperature (RT) the $Ag_2(SeTeS)_1$ chalcogenides show exceptional plastic deformability and high thermoelectric performance making them suitable materials for wearable thermoelectrics. In this study, we investigate ternary and quaternary $Ag_2(SeTeS)_1$ solid solutions with Se/Te doping closer to Ag_2S end.

A series of samples was prepared by rapid casting: $Ag_2S_{1-x}Se_x$, $Ag_2S_{1-x}Te_x$ and $Ag_2S_{0.5}Te_{0.5-x}Se_x$ (x = 0.1, 0.2, 0.3, 0.4) in to form of Ø = 3 mm, 12 mm long homogeneous ingots. Significantly high ZT = 0.47 at RT was observed for $Ag2S_{0.7}Se_{0.3}$ (so far measured by Harman method).

$Ag_2(S/Se)_1$ and $Ag_2(Se/Te)_1$ ternary systems crystalize in an orthorhombic or monoclinic structure, depending on the detailed S/Se, Se/Te atomic ratio. Addition of Te into these systems is introducing favorable cubic phase, as well observed in $Ag_2(S/Te)_1$ ternary middle region. Well known monoclinic – cubic phase transformation of $Ag_2(S/Se)_1$ [1] is pulled down to lower temperatures (even under RT) where it stabilizes with rising Te concentration. Highly disordered Ag atoms stay present in the cubic phase mimicking amorphous structure examined by diffraction.

In this contribution we present a systematic study of $Ag_2(SeTeS)_1$ chalcogenides. Temperature induced structural phase transformations are examined by differential scanning calorimetry and synchrotron in situ diffraction. Inelastic neutron scattering investigations shed light on the compounds' phonon properties showing a phonon renormalization and broadening with addition of Se/Te to Ag_2S and temperature treatment. The results are backed up by density functional theory and molecular dynamics calculations. We present data from thermoelectric characterizations carried out from RT up to 200°C.

References

[1] Bontschewa-Mladenowa, Z. and Zaneva, K. (1977), Untersuchung des Systems Ag2Se-Ag2S. Z. anorg. allg. Chem., 437: 253-262. https://doi.org/10.1002/zaac.19774370137

Small-angle neutron scattering of a pentameric ligand-gated ion channel reveals a dynamic regulatory domain

Marie Lycksell[a], Urška Rovšnik[a], Anton Hanke[a,b], Anne Martel[c], Rebecca J. Howard[a], Erik Lindahl[a]

[a]Stockholm University, Stockholm, Sweden
[b]Heidelberg University, Heidelberg, Germany
[c]Institut Laue-Langevin, Grenoble, France

marie.lycksell@scilifelab.se

Pentameric ligand-gated ion channels (pLGICs) perform electrochemical signal transduction in organisms ranging from bacteria to humans. Among the prokaryotic pLGICs there is an architectural diversity involving N-terminal domains (NTDs) not found for the eukaryotic relatives, exemplified by the calcium-sensitive channel DeCLIC. Here, we characterized DeCLIC structure using cryogenic electron microscopy (cryo-EM), small-angle neutron scattering (SANS), and molecular dynamics (MD) simulations. In both the presence and absence of calcium, cryo-EM reconstructions were similar to a previously reported calcium-bound x-ray structure. The NTDs exhibited lower local resolution than the canonical unit, consistent with these domains being relatively mobile. The small-angle scattering profile revealed a feature not explained by the available structures, indicating that further conformational diversity is available to DeCLIC. MD simulations indicated that this profile is largely attributable to rigid-body motions of the NTDs relative to the protein core, including conformations similar to those in experimental structures, as well as more expanded and asymmetric conformations. Using these expanded conformations, it was possible to fit the previously unexplained SANS feature, indicating the presence of such conformations under solution conditions. This work reveals the range of motion available to the DeCLIC NTDs, expanding the conformational landscape of the pLGIC family; and demonstrates the power of combining low-resolution, high-resolution, and simulations data in the study of protein structure.

ORSO- Open Reflectometry Standards Organisation

Tom Arnold[a], *Bridget Murphy[b,c], Jochen Stahn[d], Max Skoda[e], Brian Maranville[f], Andrew Nelson[g], Christy Kinane[e], Andrew McCluskey[a]*

[a]European Spallation Source, Sweden
[b]Institute for Experimental and Applied Physics, University of Kiel, Germany
[c]Ruprecht Haensel Laboratory, University of Kiel, Germany
[d]PSI, Switzerland
[e]ISIS Neutron and Muon Source, STFC, UK
[f]NIST, Gaithersburg, Maryland, USA
[g]ANSTO, Sydney, Australia

tom.arnold@ess.eu

ORSO is a group of reflectometry scientists from across the globe that are interested in improving our field through collaboration. ORSO is open to everyone. Our focus is on neutron and X-ray reflectometry. We will present the aims and focus of our working groups which are arranged as follows:

The education and outreach working group aims to develop understanding of reflectivity techniques and disseminate information about reflectivity analysis.

The File formats working group aims to draft a specification for a standard file format for use across X-ray and neutron reflectivity. This is an important goal to move towards interoperability across facilities.

The analysis working group is focused on cross-facility collaboration on standardized data analysis “best practices” and shared resources to make it easier to build and use reflectometry analysis software

The reproducibility working group aims to improve measurement reproducibility through the creation of informative documentation regarding experimental set-up and standardisation of instrumentation, generate general guidelines on data reduction, to ensure accurate and reproducible data and engage others in reproducible analysis, including help documentation and guides.

This organisation and its working groups are open to all, to join a working group please just contact one to the authors or go to our web page **www.reflectometry.org**. Together we can do better science.

Exploiting symmetry with brille

Gregory S. Tucker[a]

[a]*European Spallation Source ERIC*

gregory.tucker@ess.eu

The properties of crystalline materials are studied by various means including inelastic neutron scattering (INS), always with the goal of increased understanding of the material in question and often aiming to apply knowledge gained more broadly. All such properties are constrained to be periodic with the period of the crystalline lattice which typically includes translational and rotational symmetries. The periodic unit of the lattice is the irreducible Brillouin zone (irBz), and INS measurements are able to collect intensity over many such equivalent volumes of momentum-space.

Extracting the underlying physical properties of the material typically requires use of a model system, often implemented as a computer program which calculates intensities for a series of fixed positions in momentum-space. These calculations must also be periodic over the lattice and the programs typically can not easily be scaled-up to the calculate for all independent momentum-space positions probed in an INS measurement.

A computer program, brille, has been created in order to support the fitting of such models to measured INS data. It handles finding the irBz volume for a lattice, creates a space-filling grid within the identified irBz, and identifies equivalent momentum-space points within the irBz for arbitrary momentum-space points. Additionally, brille can hold user-provided data at the vertices of the space-filling grid and subsequently interpolate at arbitrary irreducible momentum-space points.

The interpolated results at a point in the irBz are not strictly identical to what a model would calculate at the equivalent momentum-space point. In these cases the results are related by the symmetry operation which relates the irreducible and non-irreducible points and, if the relationship is known and provided, brille can provide the correct results up to approximations caused by the linear interpolation for arbitrary momentum-space points. Thus far, brille supports the correct handling of the eigenvectors and eigenvalues of the grand dynamical matrix and can be used along with, e.g., Euphonic, to provide linear interpolated phonon modes and intensities from electronic structure calculations.

By interpolating model values at arbitrary momentum points, brille can lessen the work required of the model functions and improve simulation and fitting times of full INS data sets.

Spectral characterization of the SUN-2 ultracold neutron source by vertical time-of-flight

Thomas Neulinger[a], *Hanno Filter*[a,b], *Oliver Zimmer*[a]

[a]*ILL, Grenoble, France*
[b]*TU Munich, Munich, Germany*

neulinger@ill.fr

The SUN-2 source [1] at the ILL is a prototype source of ultracold neutrons (UCN) that converts cold neutrons into UCN via downscattering in superfluid helium. UCN, which have kinetic energies on the order of hundreds of nanoelectronvolts, can be trapped in material-walled storage vessels and used, for example, in experimental searches for the neutron electric dipole moment. Continued improvements to the statistical sensitivity of such measurements can be achieved at new high-flux sources, such as those planned at the ESS [2] and the ILL [3].

The spectral characterization of these sources will be instrumental to the determination of critical design factors of experiments installed there. This characterization is often pursued by measuring neutron time-of-flight (TOF) through horizontal guides. Unfortunately, this technique prohibits spectroscopy of UCN with energies below the optical potential of the detector entrance window, resulting in a design blind spot for experiments that aim to maximize their statistical reach.

This difficulty is overcome by the vertical TOF technique, in which falling UCN are accelerated to velocities sufficient to enter the detection volume. Using this method, measurements were made of the full UCN energy distribution of SUN-2 in its most recent configuration, with its internal production volume coated by a top layer of the fluoropolymer CYTOP [4]. Results are presented for both continuous and accumulation operation modes of the source. To our knowledge, this work consitutes the first measurement of the low energy tail of the UCN spectra produced at a superfluid helium based UCN source.

References

[1] A. Steyerl, *Ultracold Neutrons* (pp. 70–76), World Scientific (2020).
[2] E. Klinkby et al., Adv. High Energy Phys., vol. 2014, Article ID 241639 (2014).
[3] O. Zimmer and R. Golub, Phys. Rev. C, vol. 92, 015501 (2015).
[4] T. Neulinger, Ph.D. thesis, University of Illinois Urbana-Champaign (2021).

Neutron diffraction study of surfactant-skin lipid model interaction

Yao Chen[a], Bruno Demé[b], Jian R Lu[c], Peixun Li[a]

[a]ISIS Facility, Rutherford Appleton Laboratory, STFC, OXON, UK
[b]Institut Laue-Langevin, Grenoble, France
[c]Department of Physics & Astronomy, the University of Manchester, Manchester, UK

Yao.chen@stfc.ac.uk

Motivations and methods. Surfactants have been widely used in skin care and other related applications, our knowledge about how surfactants interact with stratum corneum (SC) lipids remains limited. This work reports how surfactants interact with a lipid SC model by neutron diffraction and molecular dynamics (MD) simulations, focusing on examining the impact of surfactant molecular architecture.[1] The surfactant-SC mixed membrane was constructed by an equimolar mixture of Ceramide/Cholesterol/Fatty acids and surfactant at 1% molar ratio of total lipids. Neutron diffraction experiments and MD simulation were performed to determine the model membrane structure, followed by characterizing the impact from surfactant binding.

Findings

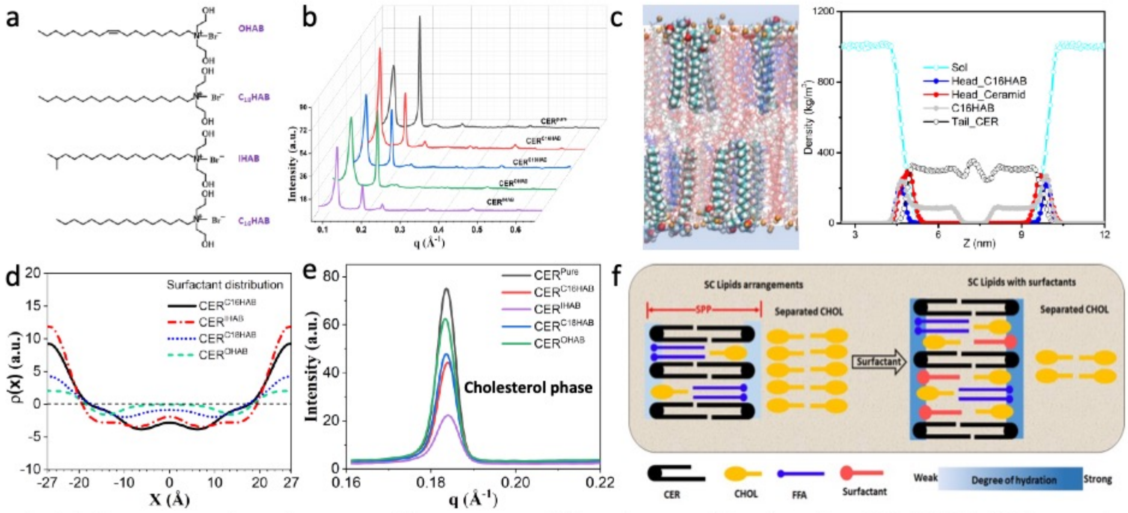


Figure 1. (a) Structure of surfactants, (b) neutron diffraction profiles for the CER/CHOL/FFA membrane with and without surfactant hydrated and measured at 100% D_2O, (c) MD simulation of the repeating units,(d) surfactant NSLD profile in the SPP structures, (e, f) schematic for the surfactant-lipid mixed model membrane, crystalline CHOL molecules migrate into the SPP with enhanced hydration of the bilayer.

Conclusions. Neutron diffraction reveals that addition of surfactant significantly increases the overall hydration of the model membrane in the hydrophilic domain and changes the cholesterol distribution. The variations strongly depend on the hydrophilicity of surfactant, resulting from the hydrophobic chain architecture. This work has offered useful insight into the roles of surfactant and CHOL in mediating hydration and local structure of the SC membrane, providing important guidance for the research and development of surfactant-based transdermal delivery systems.

References

[1] Chen, Y.; Liao, M.; Ma, K.; Wang, Z.; Demé, B.; Penfold, J.; Lu, J. R.; R. P. Webster, J.; Li, P. *J. Colloid Interface. Sci.* **2022**, 608, 405-415.

Degradation of lipid films upon nanoparticle contact studied by simultaneous neutron reflectometry, infrared spectroscopy and ellipsometry

Reiner Dahint[a], Christian Busch[a], Bela Nagy[b], Andreas Stöcklin[a], Philipp Gutfreund[c], Thomas Ederth[b]

[a]Applied Physical Chemistry, Heidelberg University, Heidelberg, Germany
[b]Division of Biophysics and Bioengineering, Dept. of Physics, Chemistry and Biology, Linköping University, Linköping, Sweden
[c]Institut Laue-Langevin, Grenoble, France

Reiner.Dahint@pci.uni-heidelberg.de

During the last decade, metal and metal oxide nanoparticles (NPs) have found increasing use in biomedical applications and consumer products. Hence, the question arises whether they may pose risks to human health [1-3]. In fact, various studies suggest that the interaction of NPs with cell membranes may affect cell function and contribute to toxic effects. Here, we utilize simultaneous neutron reflectometry (NR), infrared spectroscopy (IR) in total reflection geometry (ATR-IR) and spectral ellipsometry (SE) measurements to monitor the interaction of gold NPs (AuNPs) with oligobilayers of 1,2-dimyristoyl-*sn*-glycero-3-phosphocoline (DMPC), serving as model cell membranes, at temperatures below and above the main phase transition temperature, T_M, of the lipids.

For such combined measurements, a mobile IR and SE setup has been developed which can be interfaced to neutron reflectometers with both horizontal and vertical sample orientation. While NR allows for detailed characterization of internal film structure, IR spectroscopy provides information on molecular groups, molecular orientation and conformation. Finally, SE facilitates determination of refractive indices or large film thicknesses which are not directly accessible by NR. IR and SE are also a valuable complement to NR measurements due to their significantly higher time resolution.

In our experiments, NR, ATR-IR and SE consistently show that DMPC oligobilayers degrade upon AuNP contact. This supports the assumption that NPs may have toxic effects on biological cells. The degradation process is accelerated for temperatures above T_M, i.e. after transition from the gel-like P_{β}' to the liquid-crystalline L_{α} lipid phase. The phase state is directly monitored by ATR-IR via the position of the CH_2 stretching bands. Interestingly, the interaction with AuNPs seems to disturb the structure of both lipid phases as indicated by the shift of CH_2 bands upon AuNP exposure. Based on the observation that the difference in band position is reduced with increasing incubation time, one may conclude, that the structural features of the lipid bilayers, equilibrated below and above T_M, show increasing similarities upon AuNP interaction.

References

[1] A. Nel, T. Xia, L. Mädler, N. Li, Science, 2006, 311, 622-627.

[2] S. Shin, I. Song, S. Um, Nanomaterials, 2015, 5, 1351-1365.

[3] W. Yang, L. Wang, E. M. Mettenbrink, P. L. DeAngelis, S. Wilhelm, Annu. Rev. Pharmacol. Toxicol., 2021, 61, 269-289.

Hardening of phonons in $SrTiO_3$ in electric fields measured with neutron scattering

Henrik Jacobsen[ab], *Kristian á Brúnni*[a], *Urs Staub*[b], *Uwe Stuhr*[b], *Marek Bartkowiak*[b], *Christof Niedermayer*[b]

[a]*Niels Bohr Institute, Copenhagen, Denmark*
[b]*Paul Scherrer Institute, Villigen, Switzerland*

Henrik.jacobsen@nbi.ku.dk

$SrTiO_3$ is a quantum paraelectric, with incipient ferroelectricity, that is being suppressed by quantum fluctuations [1]. Because of the vicinity to the phase transition to ferroelectricity, the material properties of $SrTiO_3$ are strongly sensitive to external electric fields. In particular, previous measurements have shown that a soft optical phonon hardens at the Γ point in moderate electric fields [1]. In those experiments, only the Γ point could be measured, and although the dispersion of phonons in zero field is well known [2-5], it is therefore unknown how it changes with applied electric fields.

Interestingly, recent work driving the soft mode with strong single cycle THz pulses resulted in strong non-linear phonon-phonon couplings [6]. Strong anharmonicities have also lead to the disappearance of acoustic phonon branches in inelastic neutron scattering experiments. [5]. Most needed is a better understanding of the correlation between the quantum fluctuations, the octahedral rotations and the occurrence of the quantum critical point. see e.g. [7].

To address this problem, we have used inelastic neutron scattering to measure the dispersion of the soft phonons as function of electric field. We show that the phonon frequencies are most strongly influenced by the electric field near the Γ point. In addition, we find that the electric field effect depends strongly on which Brillouin Zone is being measured. In this presentation I will explore how our measurements provide new insights into the soft mode of $SrTiO_3$.

References

[1] J. M. Worlock and P.A. Fleury, PRL 19, 1176 (1967).
[2] W. G. Stirling, J. Phys. C, 5, 2711 (1972).
[3] Y. Yamada and G. Shirna, J. Phys. Soc. Jpn. 26, 396 (1969).
[4] Choudhury et al., PRB 77, 134111 (2008).
[5] X. He et al., PRL 124, 145901 (2020).
[6] Kozina et al., Nat.Phys. 15 2019, Li et al., Science 364, 1079–1082 (2019).
[7] J. M. Edge, PRL 115, 247002 (2015).

Abstract: 135

Searches for baryon number violation using HIBEAM

Matthias Holl[a]

[a]European Spallation Source ERIC, Lund, Sweden

matthias.holl@ess.eu

The HIBEAM/NNBAR program is a proposed two-stage fundamental science experiment at the European Spallation Source (ESS) to search for baryon number violation[1].

Its first step, HIBEAM, is not only intended as a stepping stone towards the full NNBAR experiment, but will also be used to search for the baryon number violating conversion of a neutron into a sterile neutron ($n \to n'$) via neutron disappearance and neutron regeneration measurements. It will also allow for initial studies into direct ($n \to \bar{n}$) and indirect ($n \to n' \to \bar{n}$) neutron-antineutron conversions via the detection of matter-antimatter annihilations.

The experiment will be situated on a fundamental physics beamline at ESS. The magnetic field within the beamline will need to be controlled, as the processes intended to be studied are highly sensitive to the external field. The HIBEAM annihilation detector will consist of a dedicated TPC currently under the development surrounded by the WASA electromagnetic calorimeter [2] as well as scintillation detectors used to veto cosmic background. For the neutron disappearance and neutron regeneration experiments, several detector options are under investigation, including 3He-detectors and solid-state scintillators.

In this talk, the physics goals of the HIBEAM experiment will be outlined and an overview over the planned beamline and detection systems will be given. The current status of development activities will also be discussed.

References

[1] Addazi *et al.* "New high-sensitivity searches for neutrons converting into antineutrons and/or sterile neutrons at the HIBEAM/NNBAR experiment at the European Spallation Source", J.Phys.G 48 (2021) 7, 070501

[2] C. Bargholtz *et al.* "The WASA Detector Facility at CELSIUS" , Nucl.Instrum.Meth. A594 (2008) 339-350

Gradual emergence of superconductivity in underdoped La$_2$-xSrxCuO$_4$

Kim Lefmann[a], Ana-Elena Tutueanu[a,b], Machteld E. Kamminga[a], Tim B. Tejsner[a,b], Henrik Jacobsen[a,c], Henriette W. Hansen[a], Monica-Elisabeta Lacatusu[a,c], Jacob Baas[d], Kira L. Eliasen[a,e], Jean-Claude Grivel[f], Yasmine Sassa[g], Niels Bech Christensen[h], Paul Steffens[b], Martin Boehm[b], Andrea Piovano[b], Astrid T. Rømer[a]

[a]Niels Bohr Institute, University of Copenhagen, Denmark
[b]Institut Laue-Langevin, Grenoble, France
[c]Laboratory of Neutron Scattering and Imaging, Paul Scherrer Institute, Switzerland
[d]Zernike Institute for Advanced Materials, University of Groningen, The Netherlands
[e]Glass and Time, IMFUFA, Department of Science and Environment, Roskilde University, Denmark
[f]Department of Energy Conversion and Storage, Technical University of Denmark, Lyngby, Denmark
[g]Department of Physics, Chalmers University of Technology, Göteborg, Sweden
[h]Department of Physics, Technical University of Denmark, Lyngby, Denmark

lefmann@nbi.ku.dk

The interplay between magnetism and superconductivity in the cuprates has been a recurring theme since the discovery of high-temperature superconductivity. We here present triple-axis neutron scattering studies of the low-energy magnetic fluctuations in underdoped archetypical cuprate La$_2$-xSrxCuO$_4$ with x=0.05, 0.06 and 0.07, providing quantitative evidence for a direct competition between these fluctuations and superconductivity. At dopings x=0.06 and x=0.07, three-dimensional superconductivity is found, while only a very weak signature of two-dimensional superconductivity residing in the CuO$_2$ planes is detectable for x=0.05.

Upon application of a strong external magnetic field, we found a surprising suppression of the low-energy fluctuations at all three dopings, in contrast to the enhancement of low-energy fluctuation found in La$_2$-xSrxCuO$_4$ with x>0.10 [1,2]. While this can be reconciled with the low doping values, our results also imply that the response of two-dimensional superconductivity (x=0.05) to a magnetic field is similar to that of a bulk superconductor (x=0.06 and x=0.07). Our results provide direct evidence of a very gradual onset of superconductivity in cuprates across the doping phase diagram.

References

[1] B. Lake et al, Science **291**, 1759 (2001)
[2] J. Chang et al, Phys. Rev. Lett **102**, 177006 (2009)

Liposomes under the influence of short-chain alcohols

Fernanda Alvarado Galindo[a,c], *Ingo Hoffmann*[a], *Joachim Venzmer*[b] *and Michael Gradzielski*[c]

[a]*Institut Laue-Langevin (ILL), Grenoble, France*
[b]*Evonik Operations GmbH*
[c]*Fachgebiet Physikalische Chemie/Molekulare Materialwissenschaften, Institut of Chemistry, Technische Universität Berlin, Germany*

alvarado@ill.fr

Phospholipid liposomes are of central importance in pharmacology and cosmetics. Typically, they are obtained by injecting a concentrated ethanolic PL solution (or other alcohol) into water. The PL solution is usually a complex mixture of phospholipids, stabilizers and active ingredients loaded into the bilayer upon self-assembly. Thus, after injection, lipids, and actives interact with solvent molecules prior to alcohol removal. Although studies suggest an increase in area per lipid molecule [1, 2], membrane fluidity and disorder [3] in the presence of alcohol, it remains unclear to which extent they behave as cosurfactants or their impact on the mesoscopic structure of the liposomes.

This study aimed to elucidate the location of short-chain alcohols in the liposomal dispersion and analyze its impact on bilayer architecture. We see an overall tendency of increased bilayer curvature with increasing alcohol content using light scattering analysis. Moreover, we exploit the contrast difference between lipids and deuterated solvents with neutron scattering experiments. For this, we prepared unilamellar liposomes in solutions containing one of the following alcohols: methanol, ethanol, propanol, or butanol. We observe increased alcohol uptake in the bilayer with longer chain length and higher alcohol concentration by small-angle neutron scattering (SANS). In addition, we used Neutron Spin Echo to resolve membrane dynamics and analyze the stiffness of the bilayer upon changes in solvent quality.

Understanding how different alcohols influence the structure and dynamics of phospholipid bilayers will render essential information to develop more stable, green and functional liposome formulations.

References

[1] J. A. Barry, K. Gawrisch. Direct NMR Evidence for Ethanol Binding to the Lipid-Water Interface of Phospholipid Bilayers. Biochemistry. 1994, 33, 8082–8088.
[2] H. V. Ly, M. L. Longo. The Influence of Short-Chain Alcohols on Interfacial Tension, Mechanical Proper- ties, Area/Molecule, and Permeability of Fluid Lipid Bilayers. Biophysical Journal. 2004, 87, 1013–1033.
[3] M. Patra, E. Salonen, et al. Under the Influence of Alcohol: The Effect of Ethanol and Methanol on Lipid Bilayers. Biophysical Journal. 2006, 90, 1121–1135.

Abstract: 138

Investigation of candidate samples for a Bragg edge neutron imaging round robin

Ranggi Ramadhan[a], Thilo Pirling[b], Winfried Kockelmann[c].

[a]Solid Mechanics Research Group, University of Bristol, Bristol, United Kingdom
[b]Institut Laue Langevin, Grenoble, France
[c] STFC-Rutherford Appleton Laboratory, Didcot, United Kingdom

ranggi.ramadhan@bristol.ac.uk

Bragg edge neutron imaging extends beyond standard neutron radiography as it is capable of non-destructively mapping important materials properties such as strain, concentrations, and crystallographic texture in the interior of materials and components. Although the number of studies is increasing steadily, Bragg edge analyses are far from being routine, and there is a need for standardized collection and analysis procedures, and for developing software to raise confidence of academic and industrial users in the new Bragg edge methods. It is proposed to address these development needs by carrying out cross-facility benchmarking of the Bragg edge method through a 'round robin' activity, as a first step towards standardisation. The aim is to benchmark the known and new capabilities on different types of instruments, to assess levels of accuracy, precision, and detection limits of Bragg edge transmission imaging for residual strain and phase composition analysis. The round robin activity is relevant for imaging beamlines such as ODIN@ESS and for 'conventional' strain scanners such as SALSA@ILL.

Such effort would require a common set of sample suitable for the particular need of Bragg edge imaging round robin activity. Geometry, size, material, the presence of residual stress fields and crystallographic texture, as well as relevance to engineering problem are among the criteria. A few samples have been identified as potential candidates for the activity, and characterisations have been carried out. Figure 1 presents a comparison of residual strains from two of the samples, i.e., steel-copper shrink-fit and laser peened steel, measured by Bragg edge imaging on IMAT, ISIS, and by monochromatic neutron diffraction on SALSA, ILL. The two measurements showed reasonable agreement. Advantages and limitations of the current set of samples will be discussed.

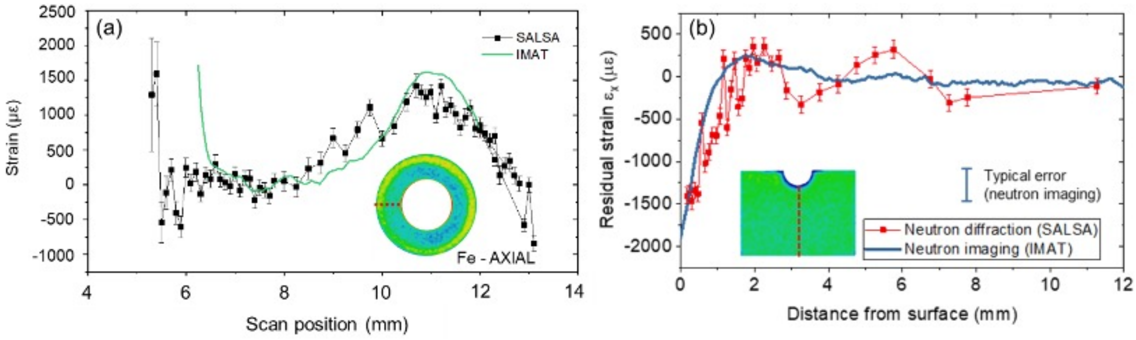


Figure 1 Comparison between residual strains measured by Bragg edge neutron imaging (IMAT-ISIS) and neutron diffraction (SALSA-ILL) on round robin sample candidates: (a) Fe part of steel-copper shrink fit, and (b) laser peened steel.

Diffusion in supramolecular gels for drug delivery

<u>Riccardo Morbidini</u>[a,b], Dr. Tilo Seydel[a], Dr. Katharina Edkins[b]

[a]Institut Laue Langevin , Grenoble , France
[b]University of Manchester, Manchester, UK

morbidini@ill.fr

The dual nature of gels, a fibrous solid network with a large, trapped liquid fraction, is attracting increasing pharmaceutical attention as they can provide sustained release of loaded drug com- pounds [1]. In supramolecular gels the solid scaffold is made from small organic molecules which can assemble through non-covalent interaction upon an external stimulus (such as temperature or pH change)[2]. Currently, there is no link between the molecular diffusion within and the macroscopic drug release from gels. To elucidate the influence of surface effects and confinement on drug self-diffusion, we are investigating a model bis-urea supramolecular gel based on water/ ethanol mixed solvent as liquid component [3][4][5] as well as a peptide supramolecular gel whose main liquid part is water[6]. By collecting QENS data we have shown how diffusion on the pico- second scale is quicker inside the gel both for the solvent[3] and for pharmaceutically interesting solute molecules[5]. Conversely, looking at longer time scales with PFG-NMR led to the opposite observation with a slowing of solvent diffusion within the gel [3]. Consequently, QENS is essential to provide a view to length scales unaffected by the confinement effect due to the fibers and take into account only surface effects, which alter the hydrodynamic environment in which the guest molecule moves. Building on this findings we recently observed, with BATS@ILL data, that in the peptide gel the effect can be the reverse, with a slowed down diffusion of the drug in the gel with respect to the bulk solvent (fig.1 left). We also explored the new opportunities given by polarized neutrons on LET@ISIS spectrometer to split the coherent and incoherent dynamic structure factors over a wide q range with sub-meV resolution. This will allow us, by selective deuteration, to resolve either the different dynamics of a D2O/ethanol-OD binary mixture (fig.1 right) or to understand whether there is any coupling between the dynamics of the protiated guest molecule and the fully deuterated water/ethanol solvent.

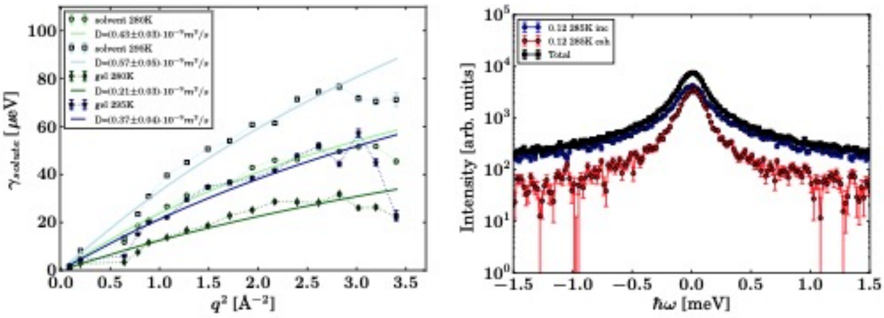


Fig.1
(left) HWHM of Ibuprofen sodium in Fmoc-FF gel, fitted with a jump diffusion model;
(right) Separation of coherent (red) and incoherent (blue) dynamic structure factors through LET polarized neutrons, and sum of the spectrum (black symbols).

References

[1] K.J.Skilling, *Soft Matter*, **10**, 237-256 (2014);
[2] J. Steed, *Chem.Soc.Rev.*, **39**, 3686-3699 (2010);
[3] T.Seydel, *Chem.Commun.*, **54**, 6340 (2018);
[4] T.Seydel, *Physical. Chem.Chem. Phys.*, **21**, 9547 (2019);
[5] R.M. Edkins, *Physical.Chem.Chem.Phys.*, **22**, 10838 (2020);
[6] A.M. Smith , *Adv. Mater. 2008*, 20, 37-41;
[7] A.Arbe, *Phys Rev Research.*, **2**, 022015 (2020).

Evolution of magnetic stripes under uniaxial stress in $La_{1.885}Ba_{0.115}CuO_4$ studied by neutron scattering

M. E. Kamminga[a], K. M. L. Krighaar[a], A. T. Rømer[a], L. Ø. Sandberg[a], P. P. Deen[a,b], M. Boehm[c], G. D. Gu[d], J. M. Tranquada[d] and K. Lefmann[a]

[a]Nanoscience Center, Niels Bohr Institute, University of Copenhagen, 2100 Copenhagen Ø, Denmark
[b]European Spallation Source ERIC, Partikelgatan 224, 84 Lund, Sweden
[c]Institut Laue-Langevin, 71 Avenue des Martyrs, CS 20156, 38042 Grenoble Cedex 9, France
[d]Condensed Matter Physics and Materials Science Division, Brookhaven National Laboratory, Upton, New York 11973, USA

kristinekrighaar@hotmail.com

The cuprate La_2-xBaxCuO_4 (LBCO) is famous for exhibiting pronounced spin-charge correlations known as stripes, which have been commonly observed in all hole-doped cuprate compounds. At a doping value of x=1/8, the stripes become especially pronounced and are often accompanied by a suppression in the superconducting critical temperature Tc.

Here we present the effect of uniaxial stress on the magnetic stripes in 0.76 g single crystal LBCO with x = 0.115, previously found to have a stress-induced enhancement in the superconducting transition temperature.

We performed a neutron experiment at ThALES (ILL) using a newly built cell for uniaxial stress up to 3 GPa. Our diffraction data show that the static stripes are suppressed by stress of 30 MPa, pointing towards a trade-off between superconductivity and static magnetism, in direct agreement with previously reported μSR measurements. Additionally our inelastic data show that some of the reduced weight in the elastic channel appears to have moved to the inelastic channel. Moreover, a stress-induced momentum shift of the fluctuations towards the typical 1/8 value of commensurability is observed.

Our experiment demonstrates the feasibility to measure inelastic data under uniaxial stress even for small crystals. Our results impose a strong constraint on the theoretical interpretation of stress-enhanced superconductivity in cuprate systems.

Abstract: 141

Suppression of magnetic order and enhancement of magnetic fluctuations in frustrated magnets from the family of iron jarosites

Peiyuan Liu[a], Mathilde B Sørensen*[a], Anders B. A. Andersen[a], Kasper S. Pedersen[a], Denis Cheptiakov[a], Nikolaj NN Andersen[a], Jan P. Embs[a], Sonja Holm-Dahlin[a], Sofie Holm-Janas[a], Machteld E. Kamming[a], Henrik Jacobsen[a], Ulla G. Nielsen[a], Kim Lefmann[a]*

both authors have contributed equally

[a] University of Copenhagen, Copenhagen, Denmark

zdj992@alumni.ku.dk

The jarosite compounds are known as ideal kagome´ antiferromagnets. Magnetism is dominated by the effects of frustration, where competing interactions make it difficult for the system to find its ground state. This may, in turn, leads to non-ordered spin liquid behavior, which has so far not been unambiguously observed. At low temperatures, the magnetic structure of the "standard" K-Fe-jarosite becomes the 120°, "q=0" magnetic structure formed by the propagation of triangular motifs.

We here study ND4-Fe-jarosite, D3O-Fe-jarosite, and H3O-Fe-jarosite, in which previous studies have reported long-range and short-ranged (spin glass) order. We characterize the samples by XRD and magnetic susceptibility. Nuclear magnetic resonance (NMR) was used to measure the molecular and chemical structure of the sample. Powder neutron diffraction was performed at HRPT (PSI) and showed the predicted behavior with suppression of the ordering temperature for ND4-Fe-jarosite and absence of long-range order in D3O-Fe-jarosite. We studied the magnetic dynamics by inelastic neutron scattering at FOCUS (PSI). For the "spin glass" D3O-Fe-jarosite, we found a substantial enhancement of the magnetic fluctuations above the spin-glass temperature. We present these data and discuss how we intend to interpret them.

References

[1] S. Blundell, Magnetism in Condensed Matter, Oxford (2001) - General textbook on magnetism
[2] S. Janas, Ph.D. thesis (2021)

Abstract: 144

Magnetic structure and spin flip transition of $MnSb_4Te_7$

Siobhan M Tobin[a], Jian-Rui Soh[b], Xin Zhang[c], Yan-Feng Guo[c], Ketty Beauvois[d], Bachir Ouladdiaf[d], Andrew Boothroyd[a].

[a]University of Oxford, Oxford, United Kingdom
[b]École Polytechnique Fédérale de Lausanne, Lausanne, Switzerland
[c]Shanghai Tech University, Shanghai, China
[d]Institut Laue-Langevin, Grenoble, France

siobhan.tobin@physics.ox.ac.uk

The family of materials $(Mn(Sb,Bi)_2Te_4)((Sb,Bi)_2Te_3)_m$ offer a smörgåsbord of topological electronic states and magnetic phenomena [1-3]. The hexagonal $MnSb_4Te_7$ is one such van der Waals material. The unit cell can be described by the *P-3m1* space group, where the Sb_2Te_3 topological layers are sandwiched between magnetic $MnSb_2Te_4$ septuple layers. Theoretical calculations indicate that different spin arrangements of the Mn magnetic sublattice can strongly influence the topology of the charge carriers in the Sb_2Te_3 quintuple layers [1]. Symmetry analysis and theoretical calculations indicate that the axion insulator state usually associated with A-type AFM order will in fact persist even when the material becomes FM ordered in the presence of an external magnetic field along the *c* axis [1]. We have conducted neutron diffraction on a single crystal of $MnSb_4Te_7$ at the D10 instrument at the ILL. Our zero field measurements are consistent with A-type AFM order as seen in the Bi equivalent compound [2,3]. With increasing field along the *c* axis, we find evidence for a canted AFM state prior to a spin flip transition occurring at ~0.15 T. The magnetic structure as a function of both temperature and external field will be discussed. We also comment on implications for the dimensionality of the magnetism. Finally we compare the magnitude and site mixing of Mn^{2+} moments to those of the Bi analogue compound.

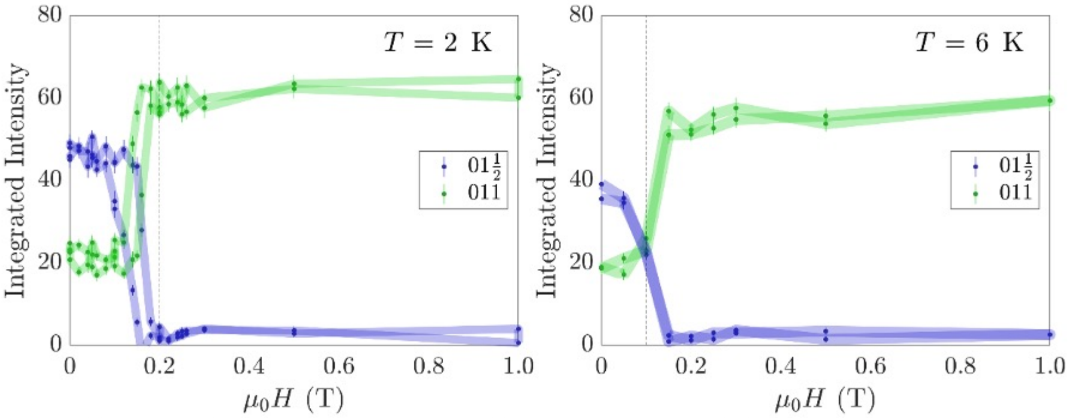


Figure: Intensity of reflections as a function of field along the c axis. A hysteresis loop opens up at a temperature of 2 K (left), which is not seen in the 6 K data (right).

References

[1] Huan, S. et al. *Physical Review Letters* **126**, 246601 (2021).
[2] Ding, L. et al. *Journal of Physics D: Applied Physics* **54**, 174003 (2021)
[3] Ding, L. et al. *Physical Review B* **101**, 020412 (2020)

Disposition of small molecules in stacked bilayers of stratum corneum lipids by neutron diffraction and selective deuteration

Christopher J. Garvey[a], Emelie J. Nilsson[b,c], Enamul Mojumdar[b,c]. Bruno Demé[d], Robert Georgii[a], Jan Holmbäck[,e,f], Vibhu Rinwa[e,f], Hwanmi Lim[e,f], Masoumeh Karimpour[e,f], Anders Carlsson[g], Carl Arevång[h], Sebastian Björklund[b,c], Johan Engblom[b,c]

[a] Technical University of Munich, Munich, Germany
[b] Department of Biomedical Science, Malmö University, Malmö, Sweden
[c] Biofilms – Research Center of Biointerfaces, Malmö University, Malmö, Sweden
[d] Institute Laue-Langevin, Grenoble, France
[e] Lipidor AB, Danderyd, Sweden
[f] Department of Materials and Environmental Chemistry, Stockholm University, Stockholm, Sweden
[g] MediGelium AB, Stockholm, Sweden
[h] Larodan AB, Solna, Sweden.

christopher.garvey@tum.de

The stratum corneum (SC) is a thin layer of anucleated cells in a lipid matrix and forms the outermost layer of skin, often depicted with a brick-and-mortar structure.[1] Understanding the molecular basis of the barrier properties of skin has important implications for diseased states such as atopic dermatitis and for topical application of therapeutics. The self-assembly of the intercellular lipid matrix forms an important aspect of the barrier properties of the SC, and it is mainly composed of ceramides, cholesterol and free fatty acids. Together, these lipids form two lamellar structures with periodicities (d) of 6 nm (short periodicity phase - SPP) and 13 nm (long periodicity phase - LPP).[2] The ceramides dictate the formation of these lamellar phases and in particular the ceramide EOS is important due to its exceptionally long structure, having an unsaturated fatty acid esterified to an ω-hydroxy fatty acyl chain. Our lipid matrix model, consisting of a 1:1:1 molar ratio of cholesterol, free fatty acids and ceramides, has a ceramide mixture of ceramide NS and ceramide EOS. In this study we have used neutron diffraction (D16 – ILL), over range of scattering vectors which span classical diffraction measurements to the range usually associated with small angle scattering to probe the localization of a small molecule, salicylic acid, within oriented lipid stacks. We used contrast variation of sorbed water to solve the phasing problem of unit cell reconstruction and provide a reconstruction of the scattering length density (SLD) profile of the lamellar/1D unit cell. By modulation of the SLD contribution of the salicylic acid to the overall profile by deuteration we are able to provide an insight of the localization of salicylic acid within the profile.

References

[1] P.M. Elias, *Epidermal Lipids, Barrier Function, and Desquamation*, Journal of Investigative Dermatology 80, (1983) S44-S49.
[2] J. A. Bouwstra, G. S. Gooris, J. A. van der Spek and W. Bras, J. Invest. Dermatol., 1991, 97, 1005-1012.

Clathrate hydrates as novel moderators for very cold neutron sources

Valentin Czamler[a,b], *Tobias Jenke*[a]
Michael Marek Koza[a], *Richard Wagner*[a], *Oliver Zimmer*[a,b]

[a]*ILL, Grenoble, France*
[b]*Universite´Grenoble Alpes, Grenoble, France*

vczamler@ill.fr

Clathrate hydrates [1] are water-based solids with large unit cells that show promise as potential moderators for use in the development of new, more intense sources of very cold neutrons (VCN). Such sources have the potential to enhance existing neutron scattering techniques, and to increase the reach of particle physics experiments that employ beams of slow neutrons.

Since their discovery in the early 19th century, clathrate hydrates have steadily gained interest in diverse fields of study. Current areas of active research, particularly in industrial chemistry, include the extraction and storage of natural gases in clathrate hydrates for energy applications. More recently, however, these materials have attracted interest as potential neutron moderators due to their low energy modes, which are a consequence of the ability of these so-called inclusion com- pounds to host guest molecules in cages that are formed by networks of hydrogen- bonded water molecules. Of particular interest is a binary clathrate hosting oxygen and tetrahydrofuran (THF) as guest molecules. The molecular oxygen provides an additional path for neutron slowdown exploiting the zero-field splitting of the magnetic triplet ground state of molecular oxygen [3].

Previous studies of these low energy modes have been carried out via measurements of the neutron dynamical structure factor $S(q,\omega)$ of several guest molecule- containing clathrate hydrates [2]. However, these investigations report $S(q,\omega)$ in arbitrary units, which cannot be used to generate quantitative predictions of the performance of clathrate hydrate moderators by simulation. We present here, in absolute units, measurements of the temperature-dependent dynamical structure factor of fully deuterated THF-containing clathrate hydrate, carried out at the Panther and IN5 instruments at the ILL. In addition, we report on time-of-flight VCN transmission measurements in THF clathrate hydrate, performed at the ILL PF2-VCN beamline. From these latter data, we extract the VCN mean free path, which is a transport parameter that is critical to the accurate simulation of potential VCN sources.

References

[1] A. K. Sum et al., Ind. Eng. Chem. Res. **48** 7457 (2009).
[2] H. Conrad et al., Physica B **350**, e647 (2004).
[3] O. Zimmer, Phys. Rev. C **93**, 035503 (2016).

Dynamics of the critical phonon modes in quantum paraelectric $SrTiO_3$

*S. Deng[a], C.R.S. Haines[a,b], D.M. Jarvis[a,c], M.J. Coak[a,d,e], A. Ivanov[b], A. Piovano[c], A. Wildes[c], and S. S. Saxena[a],**

[a]Cavendish Laboratory, University of Cambridge, J. J. Thomson Avenue, Cambridge, CB3 0HE, UK
[b]School of Physics, University of East Anglia, Norwich Research Park, Norwich, NR4 7TJ, UK
[c]Institut Laue-Langevin, 71 Avenue des Martyrs, 38000 Grenoble, France
[d]London Centre for Nanotechnology, University College London, Gower St, London WC1E 6BT, UK
[e]Department of Physics, University of Warwick, Gibbet Hill Road, Coventry CV4 7AL, UK

sd864@cam.ac.uk

We report our recently performed triple-axis inelastic neutron scattering experiments on single-crystal $SrTiO_3$ at 37 mK. These are the first measurements deep into the enigmatic quantum polar acoustic state in the vicinity of the ferroelectric quantum critical point. Measurements are taken at and around $\boldsymbol{q} = 0$ in multiple directions in reciprocal space. In addition, we explore how the pressure affects the phonon mode in $SrTiO_3$ up to 5.0 kbar around 2 K. Our observations shed light on the coupling of the soft optical mode with the acoustic phonons, and its response to external pressure [1]. We believe this could help us understand the importance of anharmonic lattice dynamics and unusual thermal transport in $SrTiO_3$.

The proximity of $SrTiO_3$ to a quantum critical point [2] and the evolution of the underlying modes when being tuned with even modest applied pressure has become a promising new branch of the study of quantum critical phenomena. The critical point here is associated with a soft optical phonon mode responsible for the ferroelectric instability through a continuous displacive transition. Our recent dielectric measurements under pressure [1,3] expose the formation of a 'quantum polar-acoustic state' below 2 K.

* This work is supported by Cavendish Laboratory and Jesus College, Cambridge, and the Department of Business, Energy and Industrial Strategy, UK (BEIS Grant Number: G115693). The experiments were carried out with the support of the Institut Laue-Langevin, Grenoble, France through the approval of beamtime for proposals 7-02-212 and 7-02-213. SD acknowledges a scholarship to pursue doctoral research from the Cambridge Trust China Scholarship Council.

References

[1] M. J. Coak, *et al.*, Phys. Rev. B **100**, 214111 (2019). [2] S. E. Rowley, *et al.*, Nat. Phys. **10**(5), 367–372 (2014). [3] M. J. Coak, *et al.*, PNAS **117**, 12707 (2020).

Abstract: 149

Unveiling Oxidation and Spin State of Fe in $Li_{1-x}Zn_xFe$

Priyanka Nehla[a,b], *Hyeyun Kim*[a], *Seda Ulusoy*[a], *Mikhail Feygenson*[c], *Peter Svedlindh*[a], *Germán Salazar-Alvarez*[a,b]

[a]*Dept. of Materials Science and Engineering, Ångström Laboratory, Uppsala University, Sweden*
[b]*Center for Neutron Scattering, Ångström Laboratory, Uppsala University, Sweden*
[c]*European Spallation Source, ESS, Sweden*

nehlapriyanka93@gmail.com

Lithium iron oxides have been studied for decades for Li-ion battery applications [1,2]. Herein, the oxidation and spin state of Fe is crucial to the magnetic properties and also to the crystal structure, as it also influences the presence of other polymorphic phases arising during the synthesis procedures. In this work, we investigate the structural and magnetic properties of $Li_{1-x}Zn_xFeO_2$ (x=0- 0.25, step=0.05) and their use for battery applications. The elemental composition has been confirmed by time-of-flight elastic recoil detection analysis (ToF-ERDA). The neutron diffraction and x-ray diffraction (XRD) patterns of the of parent compound have been refined using Rietveld refinement with cubic structure using the Fm-3m space group. The change in integrated intensity ratio of Bragg reflections [(220/111)] is attributed to enhancement in local ordering with x. Intriguingly, the magnetic ordering/transition temperature decreases with an increase in x. A magnetic irreversibility/bifurcation in zero field cooled (ZFC)- field cooled (FC) magnetization has also been found below the transition temperature for all the samples. The suppression of the ZFC-FC bifurcation and transition temperature at around 20 K under a 10 kOe dc magnetic field indicate a deviation from purely AFM interactions, which is further supported by a small hysteresis and ex- change bias in the system. The onset of a weakly frequency dependent in-phase component close the bifurcation temperature clearly indicates the presence of slow magnetic relaxation, tentatively explained by the presence of both AFM and FM interactions even though the AFM interactions are dominating. Furthermore, the presence of different Fe spin and oxidation states is discussed to understand the magnetic interactions in the system. Furthermore, Further, the ac susceptibility and time evolution of the magnetization data reveal the glassy behavior, where spin-spin correlation strength decreases with x. Moreover, local ordering in Li1-xZnxFeO2 samples have been dis- cussed by neutron pair distribution function analysis.

Acknowledgments: We thank the Swedish Research Council, VR, (Grant no. 2016-06959) for financial support. PN acknowledges the financial support from Swedish Agency for Economic and Regional Growth under the project SSRE.

References

[1] J. Luo, J. Liu, Z. Zeng, C.F. Ng, L. Ma, H. Zhang, J. Lin, Z. Shen, and H. J. Fan, Nano Lett. 13, 6136 (2013).

[2] W. Zhang, D. C. Bock, C. J. Pelliccione, Y. Li, L. Wu, Y. Zhu, A. C. Marschilok, E. S. Takeuchi, K. J. Takeuchi, and F. Wang, Adv. Energy Mater. 6, 1502471 (2016).

Molecular origins of piezoelectricity in silk

Christopher J. Garvey[a], Jacob J.K. Kirkensgaard[b], Liliana de Campo[c], Stephan Rouziere[d], Angel Alegria[e], Francesca Natali[f], Daniela Russo[f] and Vitor Sencadas[g]

[a]*Technische Universität München, Forschungsneutronenquelle Heinz Maier-Leibnitz FRM II and Physik Department E13, Lichtenbergstr. 1, 85747 Garching, Germany*
[b]*Department of Food Science, University of Copenhagen, DK-1958, Frederiksberg C, Denmark,*
[c]*Australian Centre for Neutron Scattering, Australia Nuclear Science and Technology Organization, Lucas Heights, NSW 2234, Australia*
[d]*Laboratoire de Physique des Solides, UMR 8502, CNRS, Université Paris-Saclay, 91400 Orsay, France*
[e]*Centro de Fisica de Materiales (CSIC-UPV/EHU), Materials Physics Center, San Sebastián, Spain,*
[f]*Consiglio Nazionale delle Ricerche & Istituto Officina dei Materiali c/o Institut Laue Langevin Grenoble 38042, France*
[g]*Departamento de Engenharia de Materiais e Cerâmica, Universidade de Aveiro, Portugal*

christopher.garvey@tum.de

A recent study has examined the structure of a simple piezoelectric material, isotropic mats of electro spun fibroin, suitable for biomedical and tissue engineering applications.[1] X-ray diffraction measurements indicate a material, that although crystalline, consists of many small highly strained crystallites. Small angle neutron scattering experiments have yielded a complementary perspective on fibres and the scattering from grain boundaries with solvent. These techniques lack sufficient time resolution to study the structural changes during the piezoelectric cycle, we have developed a novel approach to X-ray diffraction data acquisition which has superseded the signal to noise limitations of X-ray flux.[1] The measurements indicate, with ms time resolution, a reversible grain coarsening of silk crystallites during the piezoelectric cycle. Here we report on the dynamics of silk, and how these dynamics are activated by moisture with broad band dielectric relaxation spectroscopy, and neutron backscattering on the IN13 spectrometer (Institute Laue Langevin, Grenoble, France). Our studies indicate that these are regions of the silk fibres have dynamical characteristics in common with rubbery polymers which interact quite strongly with sorbed water. Unlike conventional amorphous regions in semi-crystalline polymers exhibiting large scale dynamics, these regions represent an energetically favorable state, which returns reversibly during the relaxation stage of the piezoelectric cycle.

References

[1] V. Sencadas, C. Garvey, S. Mudie, J.J.K. Kirkensgaard, G. Gouadec, S. Hauser, *Nano Energy*, **66,** 104106 (2019).

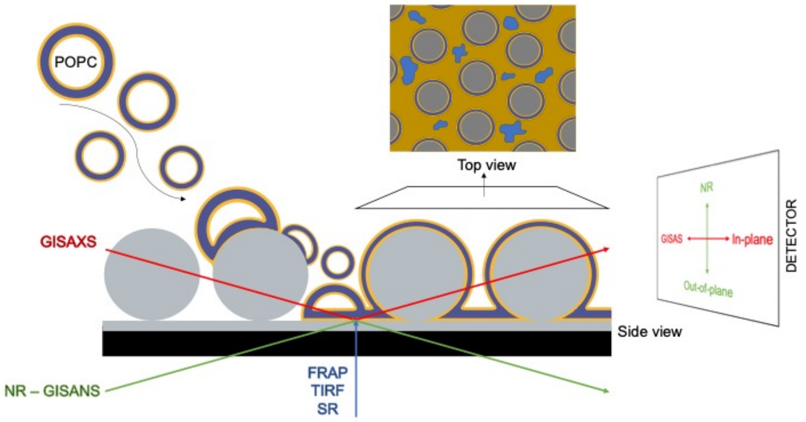
Structural characterisation of nanoparticle-supported lipid bilayers by grazing incidence X-ray and neutron scattering

Nicolò Paracini[a], Philipp Gutfreund[b], Rebecca Welbourn[c], Juan Francisco Gonzalez[a], Max Wolff[d], and
Marité Cárdenas[a,f]

[a]Department for Biomedical Science and Biofilms – Research Center for Biointerfaces, Malmö
University, Faculty of Health and Society, 205 06, Malmö, Sweden
[b]Institut Laue-Langevin (ILL), 38000 Grenoble, France
[c]ISIS Neutron & Muon Source, STFC, Rutherford Appleton Laboratory, Harwell, Oxfordshire, OX11
0QX, UK
[d]Department of Physics and Astronomy, Uppsala University, Box 516, 751 20, Uppsala, Sweden
[e]School of Biological Sciences, Nanyang Technological University, Singapore

nicolo.paracini@mau.se

The wide variety of model cell membranes available for biophysical research offer a plethora of possible approaches to study the different aspects of membrane bioscience that remain too complex to address directly on the natural cell envelope. Amongst these is the effect of membrane curvature on the functional and structural properties of the lipid and protein components of plasma membranes. There is growing interest towards the development of non-planar SLBs that deviate from canonical flat interfaces and instead display a degree of curvature of the lipid bilayer. These model systems find applications in the study of membrane curvature-mediated phenomena such as lipid and protein segregation and binding of curvature-sensitive proteins. We investigated the structure of supported lipid bilayers formed on a monolayer of spherical nanoparticles [1] using a combination of grazing incidence X-ray and neutron scattering techniques and complemented by fluorescence microscopy imaging. Ordered nanoparticle arrays assembled on a silicon crystal in a non-covalent manner using a Langmuir-Schaefer deposition were shown to be suitable and stable substrates for the formation of curved and fluid lipid bilayers that retained lateral mobility, as shown by fluorescence recovery after photobleaching. The combination of neutron scattering techniques, which grant access to sub-nanometre scale structural information at buried interfaces, and nanoparticle-supported lipid bilayers, offers a novel approach to investigate the effects of membrane curvature on lipid bilayers.



References

[1] Vorobiev A, Paracini N, Cárdenas M, Wolff M. Π-GISANS: probing lateral structures with a fan shaped beam. Sci Rep [Internet]. 2021;11(1):1–8.

Interaction of colloids with cellulose nanofibrils in aqueous solution

Constantin Harder[a,b] A. E. Alexakis[c], B. Sochor[a], A. Vagias[d], L. Porcar[e], E. Malmström[c], P. Müller-Buschbaum[b,d], S. V. Roth[a,c]

[a]DESY, Hamburg, Germany
[b]TUM, Garching, Germany
[c]KTH, Stockholm, Sweden
[d]MLZ, Garching, Germany
[e]ILL, Grenoble, France

constantin.harder@desy.de

Our goal is to fabricate colloidal photonic layers exploiting structural colours in cellulosic materials. The colloidal distribution is a result of the self-assembly of colloidal particles during the drying process after spraying deposition. [1] Therefore, the aim of our experiments is to follow the colloidal layer development during spray coating of colloidal inks used for layer formation on nanoporous templates. Cellulose nanofibrils (CNFs) are a wood-based material with a high aspect ratio (length ~500 nm and small diameter ~5 nm) and a tuneable negative surface charge. [2] The aqueous colloidal ink consists of polymer colloids with a completely deuterated and completely protonated and either hydrophobic PMMA core and a cationic hydrophilic PDMAEMA shell. In order to spray these colloids, they were diluted to a final concentration of 0.1 wt% in water [3]. The self-assembly of the colloids on CNF films in the wet layer after spray deposition is governed by the colloid-CNF-water interaction. To explore this interaction of the colloids with CNF in the dispersion state typically used for spray coating conditions, we performed small angle neutron scattering (SANS) experiments at D22 of the Institute Laue Langevin (ILL) in Grenoble. In the fully protonated colloidal suspension, the shape of the colloid nanoparticles can be modeled by a polydisperse core-shell sphere.

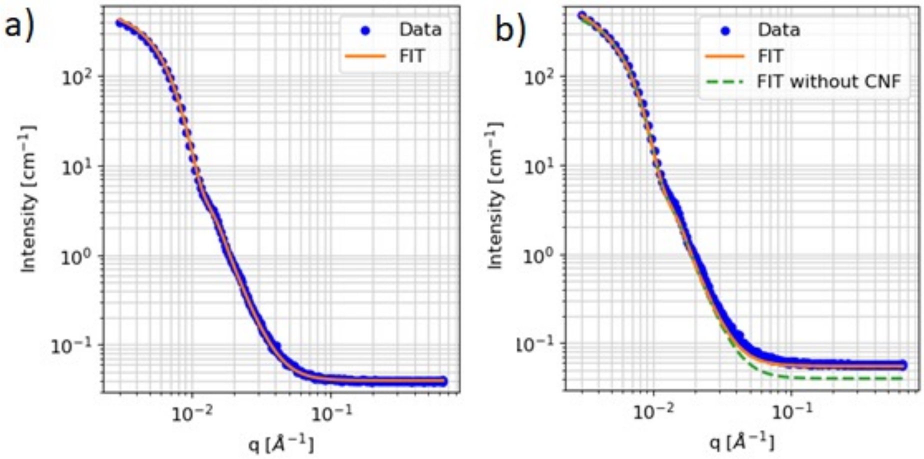


Fig.1) Protonated colloidal dispersions at $c_{\_pol}$ = 0.1 wt% in D_2O for 1410 repeat units of MMA and 25 repeat units of DMAEMA for (a) denotes single component colloidal dispersions and (b) a hybrid dispersion of colloids and CNF with the ratio 1:1. The fit is a polydisperse core-shell spherical form factor contribution without (orange) and with (green) CNF contribution.

References

[1] G. Herzog et al. *Langmuir 2013*,**29**, 36, 11260-11266 (2013)
[2] C. J. Brett, et al. *ACS Macromolecules 2019*, **52**, 12, 4721–4728 (2019)
[3] A. E. Alexakis, et al, *Green Chem.*, **23**, 5, 2113-2122, (2021)

Symmetry routes towards orbital molecules in hexagonal perovskites

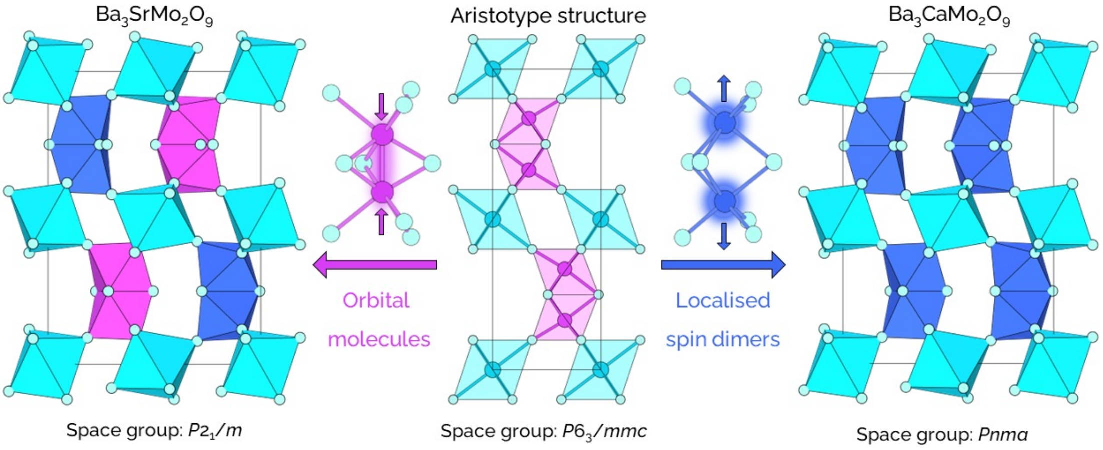
Struan Simpson[a,b] and Abbie C. Mclaughlin[a]

[a]*University of Aberdeen, Aberdeen, U.K.*
[b]*University of Warwick, Coventry, U.K.*

struan.simpson@warwick.ac.uk

In recent years, extended solids featuring closely-spaced transition metal atoms have attracted attention for their ability to form quasi-molecular electronic ground states. These "orbital molecules" arise when the overlapping metal orbitals hybridise and form direct bonds[1]. Prominent examples can be found in VO_2, in which V–V bonds form below a metal-insulator transition[2], and magnetite (Fe_3O_4), in which a complex network of three-site Fe^{3+}–Fe^{2+}–Fe^{3+} trimerons emerges[3]. However, competing interactions – such as intraatomic Hund's coupling or magnetic superexchange interactions – can preclude the formation of orbital molecules and instead favour low-dimensional magnetic ground states. Hence, it is of interest to identify mechanisms by which this competition can be overcome to generate more exotic electronic phenomena.

We have recently reported two new hexagonal perovskites of the form $Ba_3B'Mo_2O_9$ (B' = Ca, Sr)[4]. These materials contain face-sharing Mo_2O_9 geometries which impose direct overlap between the Mo^{5+} (t_{2g}^1) orbitals. However, our neutron diffraction measurements have revealed that the B' = Ca composition features localised spin dimers, while the B' = Sr analogue features a mixture of localised dimers and orbital molecules. With the aid of symmetry-mode analysis, we rationalise the dissimilar ground states of these materials based on their inequivalent octahedral tilting distortions, identifying sources of structural coupling that may be exploited to engineer orbital molecules within the hexagonal perovskite architecture as well as other extended solids.



References

[1] J. P. Attfield, *APL Mater.*, 2015, **3**, 041510.

[2] J. B. Goodenough, *J. Solid State Chem.*, 1971, **3**, 490-500.

[3] M. S. Senn *et al.*, *Nature*, 2012, **481**, 173-176.

[4] S. Simpson *et al.*, *Phys. Rev. Mater.*, 2022, **6**, 024401; S. Simpson *et al.*, 2022, Under review.

Abstract: 156

Nested mirror optics – towards efficient neutron delivery from compact high-brilliance sources

Christoph Herb [a,c], *Oliver Zimmer* [b], *Robert Georgii* [a], *Peter Böni* [c]

[a]*Heinz Maier-Leibnitz Zentrum, Garching, Germany*
[b]*Institut Laue-Langevin, Grenoble, France*
[c]*Technical University of Munich, Garching, Germany*

christoph.herb@frm2.tum.de

Upcoming high-brilliance neutron moderators at the ESS and elsewhere have raised the question how to efficiently extract and transport neutrons from such compact sources while maximizing the brilliance transfer to an instrument. Nested mirror optics (NMO) offer a viable and flexible solution for this task by circumventing the under-illumination associated with long neutron guides. A basic version, consisting of an assembly of short elliptic mirrors, is able to image a well-defined volume of beam phase space from the moderator surface onto a target, e.g., a sample or a virtual source. In contrast to neutron guides, each of the NMO's individual mirrors reflects neutrons only within a narrow range of finite angles. Due to its geometrically well-defined reflection kinematics, one can tailor the transported beam divergence and spectrum to the needs of an experiment by design of the NMO. The device thus provides a clean beam without depending on optical elements close to the source or the target, which leads to a number of practical advantages.

Besides a presentation of the concept, we will report on experimental results obtained at the multi-purpose instrument MIRA at FRM2 with an elliptic NMO prototype, see Fig. 1. We will further present preliminary results from a recent experiment at the BOA beamline at PSI, which included, among others, the imaging of a grid as shown in Fig. 2. Supported by McStas simulations, we will highlight various potential applications of NMO for neutron scattering and fundamental physics.

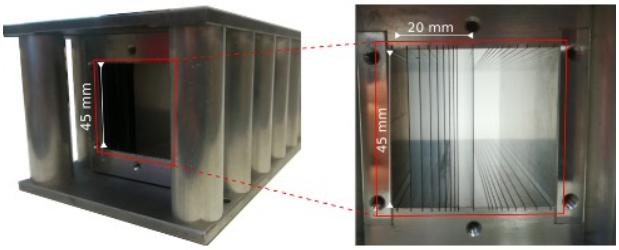


Figure 1: NMO prototype investigated at MIRA consisting of eight elliptic mirror plates. A central ninth plate facilitated the alignment process. The grooves visible on the right-hand side were left empty.

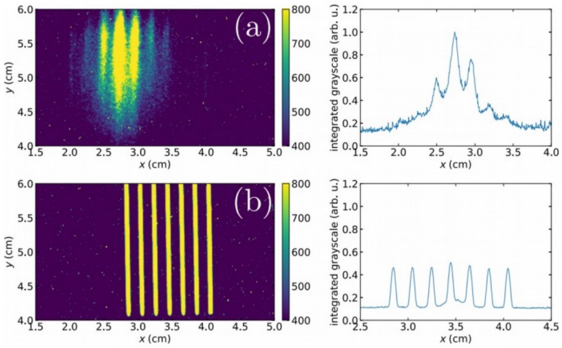


Figure 2: Imaging of a grid-like geometry by an elliptic NMO focussing in horizontal direction. (a) shows the image as obtained at the second focal point of the elliptic NMO: (b) shows the original grid as located in the first focal point Figures to the right show the vertically integrated data, respectively.

Effects of layer thickness ratio on crystalline quality of CrBx/TiBy superlattice neutron mirrors grown on 4H-SiC(0001) and Al2O3(0001)

Samira Dorri[a]*, Naureen Ghafoor*[a]*, Fredrik Eriksson*[a]*, and Jens Birch*[a]

[a]*Thin Film Physics Division, Department of Physics, Chemistry and Biology (IFM), Linköping University, Linköping, Sweden*

samira.dorri@liu.se

Neutron scattering techniques are limited by the relatively low flux of useful neutrons delivered to the sample. Improving the performance of key neutron optical components is an effective way to increase this flux. Supermirror Fermi choppers provide an attractive solution to the low flux problem at pulsed sources e.g., the ESS. The concept has been confirmed although with much lower neutron flux transmission than expected, caused by insufficient reflectivity of the supermirrors. Thus, multilayer mirrors with better layer definition than available today are needed. To achieve the ultimately flat and sharp interface definition (±1⁄2 atomic layer), it is required that the mirror is made as a single crystal, artificially layered, heteroepitaxial structure (superlattice, SL). Here we explore CrB_2/TiB_2 (0001)/Al_2O_3 (0001) and CrB_2/TiB_2 (0001)/4H-SiC (0001) SL mirrors, grown by Magnetron Sputter Epitaxy, as a solution for the compact slit package (~20×30 mm2) that is required for supermirror Fermi choppers.

CrBx/TiBy SLs with periodicities, Λ, in the range of 1-10 nm and TiB_2 thickness-to-Λ ratios, Γ, in the range of 0.2-0.8, are successfully grown using CrB_2 and TiB_2 sources, as confirmed by HRTEM, XRD and XRR. However, B-segregation due to over-stoichiometry (y>2) in TiBy, as determined by ERDA and EELS, is shown to deteriorate the structural quality. By co-sputtering of Ti and TiB_2 and varying the relative powers to the Ti and TiB_2 magnetrons (PTi = 0–50 W while keeping the total power constant $PTiB_2$ + PTi = 120 W), it is seen that at PTi/$PTiB_2$ = 20 W/100 W, a composition close to stoichiometry is achieved leading to a considerable improvement in the SLs structure and enhanced XRR peaks. The effects of the relative thickness of the TiBy and CrBx layers on the SL structures grown on 4H-SiC and Al_2O_3 substrates are highlighted. We show that 4H-SiC lattice matched substrates lead to a considerably improved crystal quality and interface roughness. Model calculations show that CrB_2/TiB_2 SLs may reach neutron reflectivities as high as 80% for just 50 deposited periods if the two isotopes 10B and 11B are localized to the TiB2 and CrB2 layers, respectively.

Abstract: 160

Correlation of superconducting and morphological properties via polarized neutron tomography

U. Oji[a], N. Kardjilov[b], I. Manke[b], A. Hilger[b], R. Cubitt[a]

[a]Institut-Laue Langevin, Grenoble, France

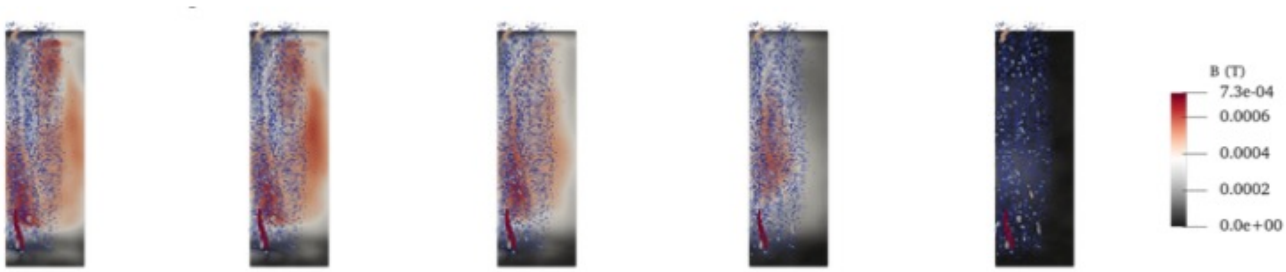
[b]Helmholtz Zentrum Berlin, Berlin, Germany

oji@ill.fr

Magnetic vortex penetration and trapping in type II superconducting materials allow their use as key components in cryomagnets. A parameter of merit to determine the performance of these superconductors (SC) is the maximum magnitude of trapped field that can exist in the material bulk. This value is dependent on a variety of factors such as the material microstructure, morphology, defect concentration and the concentration and efficiency of the pinning centres[1]. Hitherto methods of studying the effects of these material properties on this performance parameter have involved cutting of the material into smaller pieces for study. Evidently, the ability to non-destructively investigate properties in an SC bulk not only facilitates their application in technologies like cryomagnets but also scientific applications such as studying material property effects on superconducting properties.

We report on the use of neutron tomography to achieve this non-destructive characterization of superconductors specifically Yttrium Barium Cuprate (YBCO). Magnetic field is trapped in the bulk of the sample via field cooling to a temperature of 90.5 K in an external field of 0.8 mT. Using Polarized Neutron Tomography , this field distribution is reconstructed (via TMART algorithm)[2] while increasing the temperature by steps of 0.1 K until 90.9 K where the sample becomes mostly non-superconducting. The decay of the internal field is correlated with the morphology of the sample using a reconstructed volume obtained through neutron attenuation tomography which shows regions of porosity in the sample bulk volume.

The results indicate a correlation between the sample porosity and field trapping. Specifically the field is preferentially trapped in the porous regions. This is most likely due to an increase in pinning centres for the magnetic vortices around the pores. In addition, the field that remains within the pores are maintained by the super currents circulating around them. These results demonstrate nicely the potential of polarized neutron tomography for the investigation of the effects of material properties on the superconducting properties without the need to destroy the material under investigation.



References

[1] R.P Huebener, Magnetic Flux Structures in Superconductors. Springer 2001

[2] A. Hilger et al. „Tensorial neutron tomography of three-dimensional magnetic vector fields in bulk materials“. Nature Communications (2018)9:4023

Abstract: 164

Research and tuning of Y-type hexaferrites

Oskar Stepancic[a], Lingjia Shen[a], Annika Stellhorn[a], Elizabeth Blackburn[a]

[a]Department of Synchrotron Radiation Research, Lund University, Sweden

oskar.stepancic@sljus.lu.se

Magnetoelectric multiferroics are materials where ferroelectric and magnetic order parameters coexist and are coupled [1]. This makes them interesting for a variety of uses like magnetic sensors, transformers and gyrators and tuneable microwave devices [2]. Since the magnetoelectric effect appears at room temperature only for a small number of materials, there is a worldwide effort to improve our understanding of this behaviour and how to control the properties. The Y-type hexaferrite $Ba_{2-x}Sr_xMg_2Fe_{12}O_{22}$ exhibits a giant magnetoelectric effect at room temperature [3]. At present, only limited information is available on certain doping values and in relatively small applied fields. Here I will present my work on the effects of doping on the magnetic order in the hexaferrites.

References

[1] T. Kimura, *Magnetoelectric Hexaferrites*, Annu. Rev. Condens. Matter Phys. 3, 93 (2012).

[2] C.-W. Nan *et al.*, *Multiferroic magnetoelectric composites: Historical perspective, status, and future directions*, J. Appl. Phys. **103**, 031101 (2008).

[3] K. Zhai *et al.*, *Giant magnetoelectric effects achieved by tuning spin cone symmetry in Y-type hexaferrites*, Nature Communications **8**, 51 (2020).

Does oxidative damage enhance or hinder peptide insertion into lipid monolayers?

Beatrice Barletti[a], *Katherine Thompson*[b], *Adrian Rennie*[c]

[a]*Institut Laue-Langevin, Grenoble, France*
[b]*Department of Biological Sciences and Institute of*
Structural and Molecular Biology, Birkbeck University of London, London, United Kingdom
cDisciplinary Domain of Science and Technology, Physics, Department of Physics and
Astronomy, Uppsala University, Uppsala, Sweden

barletti@ill.fr

The insertion of peptides and proteins into cell membranes is crucial for a huge range of biological functions. Since real biological membranes are continuously subject to oxidative damage, in this work we study how the interaction of the antibacterial peptide protegrin with lipid monolayers changes upon partial oxidation of the membrane. Previous work has shown that oxidation of lipids in membranes leads to a substantial change in membrane structure and electrostatic interactions, as predicted from simulations [2],[3].

In this study we used neutron reflection (Figaro, ILL) to investigate how the oxidized lipid 1-palmitoyl-2-(9'-oxo-nonanoyl)-sn-glycero-3-phosphocholine, POnPC, formed from the oxidation of the unsaturated lipid 1-palmitoyl-2-oleyl-sn-glycero-3-phosphocholine, POPC, in phosphocholine based lipid monolayers at the air-water interface changes the nature and degree of interaction with the peptide protegrin. Monolayers were formed on a Langmuir trough and the peptide was injected into the subphase. The results indicate that at low surface pressures the presence of oxidized lipid allows greater insertion of the peptide into the lipid layer, but at high surface pressures this is not observed. Future work will look at monolayers that include phosphoglycerol lipids, in addition to phosphocholine lipids, as they are commonly found in bacterial membrane.

These results provide a better understanding of how oxidative damage can change the interaction of peptides with different types of lipid monolayers and enable a more realistic model of peptide:membrane interactions to be developed.

References

[1] Lad, Birembautet al., Biophysical Journal, 92, 3575 (2007)

[2] Khabiri et al., Chemical Physics Letters, 519-520, 93 (2012) [3] Thompson et al., Langmuir, 29, 4594 (2013)

Simplifying diffraction data analysis with EasyDiffraction

Andrew Sazonov [a], *Simon Ward* [a] *and Piotr Rozyczko* [a]

[a] *European Spallation Source ERIC, P.O. Box 176, SE-221 00, Lund, Sweden*

andrew.sazonov@ess.eu

Diffraction is a key tool for structure analysis. However, currently available software for modelling and analysis of diffraction data may be, on the one hand, difficult for new users looking to apply diffraction to their field of expertise and, on the other hand, not flexible enough for domain experts.

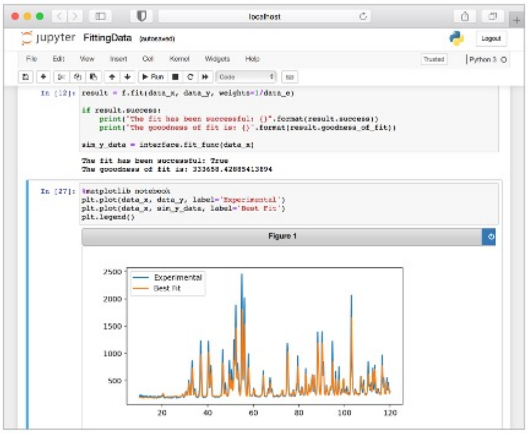
EasyDiffraction (https://easydiffraction.org) aims to lower the barrier of entry to diffraction data analysis by providing an intuitive and user-friendly graphical interface (see Figure, left), which is distributed as an all-in-one package that includes all dependencies and can be installed with just a few clicks on different operating systems. For more complex problems and increased flexibility the Python library behind EasyDiffraction can be used through Jupyter notebooks (see Figure, right) and scripting.

EasyDiffraction is built on the EasyScience framework (https://easyscience.software), a platform aimed at unifying neutron scattering analysis software. In addition to diffraction, this framework has been successfully applied to reflectometry. QENS will also be targeted in the future.

Currently EasyDiffraction has the basic features of CrysPy (https://github.com/ikibalin/cryspy) and CrysFML (https://code.ill.fr/scientific-software/crysfml) crystallographic libraries. We are collaborating with LLB and ILL regarding the CrysPy and CrysFML, respectively, and more functionality will become available as the project matures. EasyDiffraction is being developed free and open source, and we hope to attract interested people to jointly contribute to this project and help us, for the benefit of everyone, in making diffraction data analysis and modelling easier.

No.	Label	Value	Error	Fit
32	D1A@ILL wavelength	1.9120 Å		☐
33	D1A@ILL u	0.1447		☐
34	D1A@ILL v	-0.4252		☐
35	D1A@ILL w	0.3864		☐
36	D1A@ILL x	0.0000		☐
37	D1A@ILL y	0.0961		☐
38	D1A@ILL zero_shift	0.1546 deg		☑
39	D1A@ILL scale	1.4745		☑

-0.3827 -0.4677



```
In [12]: result = f.fit(data_x, data_y, weights=1/data_e)

         if result.success:
             print("The fit has been successful: {}".format(result.success))
             print("The goodness of fit is: {}".format(result.goodness_of_fit))

         sim_y_data = interface.fit_func(data_x)

         The fit has been successful: True
         The goodness of fit is: 333658.42885413894

In [27]: %matplotlib notebook
         plt.plot(data_x, data_y, label='Experimental')
         plt.plot(data_x, sim_y_data, label='Best Fit')
         plt.legend()
```

Neutrons for Europe
USER MEETING
2022

Abstract: 167

Understanding the magnetic dynamics of the strongly frustrated magnet Gd3Ga5O12 using neutron scattering and the Langevin simulation program CLaSSiC

Emma Y. Lenander[a], Simon K. K. Ørgaard[a], Frederik A. S. Philipsen[a], Ian B. McKenzie[a], Timo Vinke[a], Oleg A. Petrenko[b], Robert Bewley[c], David Voneshen[c], Elsa L'hotel[d], Pascale P. Deen[a,e], Kim Lefmann[a], Henrik Jacobsen[a,f]

[a]Niels Bohr Institute, Copenhagen, Denmark
[b]Department of Physics, University of Warvick, UK
[c]ISIS facility, Rutherford Appleton Laboratory, UK
[d]CNRS, Grenoble, France
[e]ESS, Lund, Sweden
[f]Paul Scherrer Institute, Villigen, Switzerland

Henrik.jacobsen@nbi.ku.dk

Magnetic frustration suppresses conventional long range order, allowing more exotic phases to emerge. One example is the director state in $Gd_3Ga_5O_{12}$ (GGG) [1], where clusters of spins ("directors") become correlated over long distances, despite the individual spins only forming short range correlations. The magnetic dynamics of GGG span several orders of magnitude in energy scale, and are not yet fully understood.

We have measured the magnetic dynamics of the similar compound $Gd_3Al_5O_{12}$ (GAG) in applied magnetic fields using inelastic neutron scattering [2]. The experiment was carried out on an isotopically enriched powder of GAG on the time of flight spectrometer LET.

In large fields, the ground state of GAG is strongly polarized, and the excitations can be well understood using linear spin wave theory. However, as the field is lowered, the spins become disordered, and linear spin wave theory no longer applies. In particular, a puzzling soft mode appears at zero field, where director correlations are also present.

To understand the magnetic dynamics in GAG, we have developed a general numerical Langevin simulation tool, CLaSSiC. The code has been verified against spin wave theory, and can easily simulate up to 10x10x10 unit cells of GAG. We show that the dynamics cannot be understood solely in terms of nearest neighbour exchange interactions. Rather, a combination of dipolar interactions and local anisotropies are required.

References

[1] J. A. M. Paddison *et al.*, Science **350**, 179 (2015)
[2] H. Jacobsen *et al.*, PRB **104**, 054440 (2021)

Symmetry protected 1D chains in mixed-valence iron oxides

Denis M. Vasiukov[a], Ghanashyam Khanal[b], Ilya Kupenko[c], Georgios Aprilis[d],
Sergey V. Ovsyannikov[e], Stella Chariton[e], Valerio Cerantola[d], Vasily Potapkin[e],
Aleksandr I. Chumakov[d], Leonid Dubrovinsky[e], Kristjan Haule[b], and Elizabeth Blackburn[a]

[a]Department of Physics, Lund University, Sweden
[b]Department of Physics and Astronomy, Rutgers University, USA
[c]Institut für Mineralogie, University of Münster, Germany
[d]ESRF, The European Synchrotron, France
[e]Bayerisches Geoinstitut, Universität Bayreuth, Germany

denis.vasiukov@sljus.lu.se

Iron oxides are normally Mott insulators with the only notable exception of magnetite (Fe_3O_4), which has good electrical conductivity due to the itinerant mixed-valence state of iron. During the last decade of high-pressure research a whole new series of iron oxides with the same feature was discovered, like Fe_4O_5, Fe_5O_6, Fe_7O_9 etc. All of them have closely related structures which are composed of two basic building blocks of iron polyhedra, the one-dimensional (1D) chains of trigonal prisms and two-dimensional slabs of octahedra [1]. We will present a unified crystallographic approach to the series and show that magnetic interactions in the chains have a symmetry protection against perturbations from surroundings. This leads to peculiar magnetic structures with coexisting ordered slabs and disorder chains as evidently demonstrated by Mössbauer data. Using dynamical mean-field theory calculations we found that the electrical conductivity in these oxides is strongly dependent on the type of magnetic structure. In the completely ordered magnetic state the conductivity becomes highly anisotropic reaching maximum values in the direction along the 1D chains. We will show that due to this feature these oxides can potentially host the extensively studied in theory but hardly realizable models of coupled 1D wires [2]. Wide range of possible ionic substitutions and the fact that these oxides are recoverable make them a promising platform for engineering different physical systems with variety of magnetotransport phenomena.

References

[1] Bykova, E. et al. Structural complexity of simple Fe_2O_3 at high pressures and temperatures. *Nat. Commun.* 7 (2016).
[2] Teo, J. C. & Kane, C. From Luttinger liquid to non-Abelian quantum Hall states. *Phys. Rev. B* 89, 085101 (2014).

Magnetism in the distorted Kagome-lattice intermetallic RAgGe family

C. B. Larsen[a], *O. Zaharko*[a], *D. G. Mazzone*[a] , *P. C. Canfield*[b], *S. L. Bud'ko*[b]

[a]*Paul Scherrer Institute, Villigen, Switzerland*
[b]*Ames Laboratory and Department of Physics and Astronomy, Iowa State University, Ames, Iowa, USA*

camilla.larsen@psi.ch

Kagome antiferromagnets are well-established model systems in theoretical condensed matter research. Experimental verification of theoretical solutions is, however, lacking, as real systems obeying strict conditions of the ideal Kagome lattice are sparse. In the RAgGe family of compounds, frustrated magnetism is induced by the distorted Kagome arrangement of the rare earth R+3 ions with strong magnetic easy-axis anisotropy. The physics is further enriched by itinerant electrons resulting in a wide range of exotic magnetic behaviour across the RAgGe family; HoAgGe hosts a Kagome spin-ice [1], YbAgGe reveals strong Kondo interaction and is located in a critical regime between long-range order of localised moments and non-Fermi-liquid [2], and TmAgGe exhibits metamagnetic transitions [3].

Here we present single crystal neutron diffraction results for the R = Yb and Tm compounds [4, 5]. We will discuss the work we have put into investigating topological and multi-k features as well as the final refined magnetic structures.

Work in Villigen is supported by the Swiss National Science Foundation (Grant No. 200020-182536), work in Ames is supported by the U.S. Department of Energy, Office of Basic Energy Science, Division of Materials Sciences and Engineering under Contract No. DE-AC02-07CH11358.

References

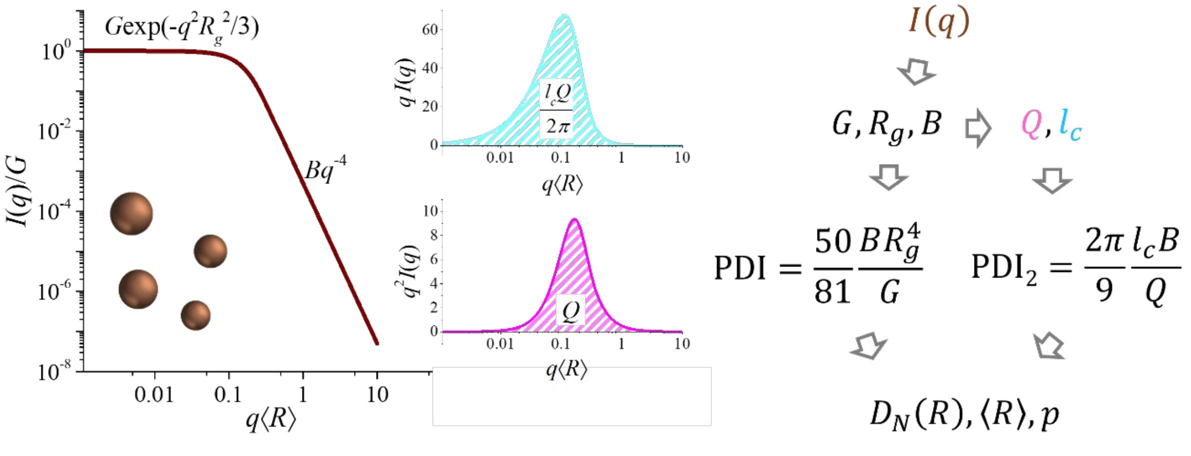
[1] K. Zhao et al, Science 367 1218 (2020)
[2] Y. Tokiwa et al, Phys. Rev. Lett. 111 116401 (2013)
[3] E. Morosan et al, Phys. Rev. B 71 014445 (2005)
[4] C. B. Larsen et al, Phys. Rev. B 104 054424 (2021)
[5] in preparation

Particle size polydispersity analysis based on a unified exponential/power-law approach to small-angle neutron scattering

Oleksandr Tomchuk[a,b]
[a]The Henryk Niewodniczański Institute of Nuclear Physics, Polish Academy of Sciences, Kraków, Poland
[b]Taras Shevchenko National University of Kyiv, Kyiv, Ukraine.

oleksandr.tomchuk@ifj.edu.pl

The use of small-angle neutron scattering in the complex analysis of particulate systems brings a number of benefits in understanding various physicochemical processes since the method is capable of providing a precise structural description at the supramolecular level. Such a description must contain information about polydispersity. There are many approaches to determine the size distribution function, $D_N(R)$. Nevertheless, they all contain certain integral equations, which can introduce additional mathematical artifacts. Beaucage *et al.* proposed an alternative approach [1]. They took into account that each of the parameters of the Guinier and Porod equations is proportional to some moment of the size distribution function, R^n. This formed the basis for an approach that makes it possible to analytically calculate the parameters of a lognormal distribution based on a dimensionless combination of parameters, designated as PDI. In this paper, we consider the possibility of using additional scattering invariants, such as the Porod integral or the correlation length, to construct an additional polydispersity index containing lower distribution moments compared to the classical PDI. This makes it possible to analytically determine the parameters of polydispersity functions other than lognormal. The cases of normal, Boltzmann, uniform, triangular, Schultz and exponential distributions, as the most frequently encountered in practice, are considered in detail.



References

[1] Beaucage G. et al., J. Appl. Cryst. 37 (2004) 523.

Kitchen-Based Light Tomography – an educational scanner for training the next generation of neutron and X-ray tomography users

Emanuel Larsson[a], Doğa Gürsoy[b], Stephen A. Hall[a]

[a]Division of Solid Mechanics, Lund University, Lund, Sweden
[b]Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA

emanuel.larsson@solid.lth.se

Kitchen-Based Light Tomography (KBLT) [1,2] is a home-made tomography scanner for educational purposes, using normal visible light. KBLT can be used as a teaching and training tool in tomography, prior to a real neutron or X-ray tomography experiment, including image acquisition, slice reconstruction, using e.g., TomoPy, and consecutive image processing and analysis, using Python packages, and 3D-rendering. KBLT resembles the setup of neutron and X-ray microtomography, where a Raspberry Pi (RPi) single-board computer is used to control a step motor (rotation stage) and a USB web camera, via a Graphical User Interface (GUI). However, instead of using X-rays or neutrons, a strong LED flashlight is used as a light source. To demonstrate the capabilities of KBLT, both non-transparent and transparent objects were scanned under both static (3D) and dynamic (4D) acquisition modes, also in combination with sample environments, thus modifying the sample during scanning.

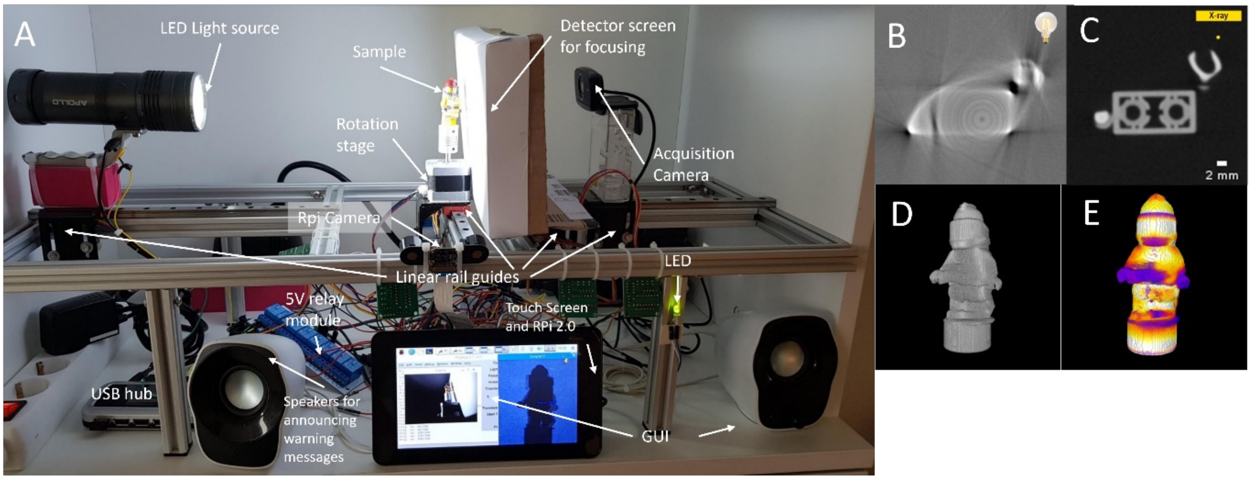


Figure 1 A) The KBLT setup, reconstructed slice from B) KBLT and C) X-ray microCT and D-E) 3D-renderings of B).

References

[1] E. Larsson, D. Gürsoy, S. A. Hall, Kitchen-Based Light Tomography – a home-based educational tool kit for training the next generation of tomography users of Large-Scale Research Infrastructures, *manuscript submitted to IEEE Access, IEEE Education Society Section on the 9th of June 2022.*

[2] From kitchen tomography to a cutting edge neutron imaging beamline, LINXS, October, 2021, Link: https://www.linxs.se/news/2021/10/11/from-kitchen-tomography-to-a-cutting-edge-neutron-imaging-beamlinenbsp

Abstract: 176

Probing the effect of ligand non-innocence on magnetic excitations in pyrazine-based metal-organic framework compounds

W. Wan[a], L. Voigt[b], H. Chen[b], M. Kubus[b], A. R. Wildes[c], H. C. Walker[d], T. Olsen[a],
K. S. Pedersen[b], N. B. Christensen[a]

[a]Department of Physics, Technical University of Denmark, Kongens Lyngby, Denmark
[b]Department of Chemistry, Technical University of Denmark, Kongens Lyngby, Denmark
[c]Institut Laue-Langevin, Grenoble, France
[d]ISIS Facility, Rutherford Appleton Laboratory, Didcot, United Kingdom

nbch@fysik.dtu.dk

In this contribution, we investigate the magnetic properties of a class of pyrazine-based metal- organic framework compounds displaying a tendency towards ligand non-innocence, i.e. the transfer of electrons from a metal ion to the ligand molecule. The electron transfer causes pyrazine to carry S=1/2, and dramatically affects electronic and magnetic properties. This is illustrated by contrasting conducting, ferrimagnetic $CrCl_2(pyz)_2$ [1] displaying ligand non-innocence with insulating, antiferromagnetic $CrI_2(pyz)_2$ [2] which does not. We have used time-of-flight neutron spectroscopy at ISIS to probe the magnetic excitation spectra of these compounds and find intense conventional antiferromagnetic spin waves in $CrI_2(pyz)_2$ but only very weak signatures of low- energy ferromagnetic modes in $CrCl_2(pyz)_2$ [3].

To directly probe the presence of a magnetic moment on pyrazine, we have investigated $GaCl_2(pyz)_2$ which contains no 3d or 4f elements. Magnetic susceptibility measurements on this compound nevertheless indicates antiferromagnetically correlated S=1/2 moments but no magnetic order above 2 K [2]. Using the D7 diffuse scattering spectrometer at ILL, we detect paramagnetic scattering from S=1/2 entities extending over a length scale consistent with the spatial ex- tent of a pyrazine ligand molecule [3].

References

[1] K. S. Pedersen et al, Nature Chemistry 10, 1056 (2018)
[2] L. Voigt. PhD thesis, Department of Chemistry, Technical University of Denmark (2020).
[2] W. Wan, PhD thesis, Department of Physics, Technical University of Denmark (2021).

YMIR: A controls integration neutron-free instrument

Stefanos Athanasopoulos[a] *and Tobias Richter*[a]

[a]*European Spallation Source (ESS) ERIC, Lund, Sweden*

stefanos.athanasopoulos@ess.eu

The control of all instrument-related hardware and components at the European Spallation Source (ESS) ERIC will be realised with the NICOS scientific instrument control software1. The communication of the various equipment with the different instrument-specific NICOS instances will be established through the EPICS system control protocol2, over private instrument-specific virtual networks. To enable a holistic control of an experiment, the objective at ESS is to also integrate non-instrument-specific sample environments into NICOS, both ESS- and user-provided. To that end, a neutron-free instrument has been developed at ESS, YMIR, for device testing and integration at an instrument-like environment (Fig. 1a). YMIR is also being used to establish the infrastructure for the recording and time-stamping of all the relevant to an experiment data and information. Using the Apache Kafka event streaming platform3, all data will be streamed and compiled into NeXus format data files4, whilst synchronised with ESS' global timing system. To demonstrate the work that will be carried out in the future with the relevant groups of the different currently-under-construction ESS instruments, an optical light tomography setup is being developed at YMIR (Fig. 1b), based on the work of Larsson et al. (2022)5. Herein, YMIR's optical light tomograph is presented, focussing mainly on the details related to its controls integration.

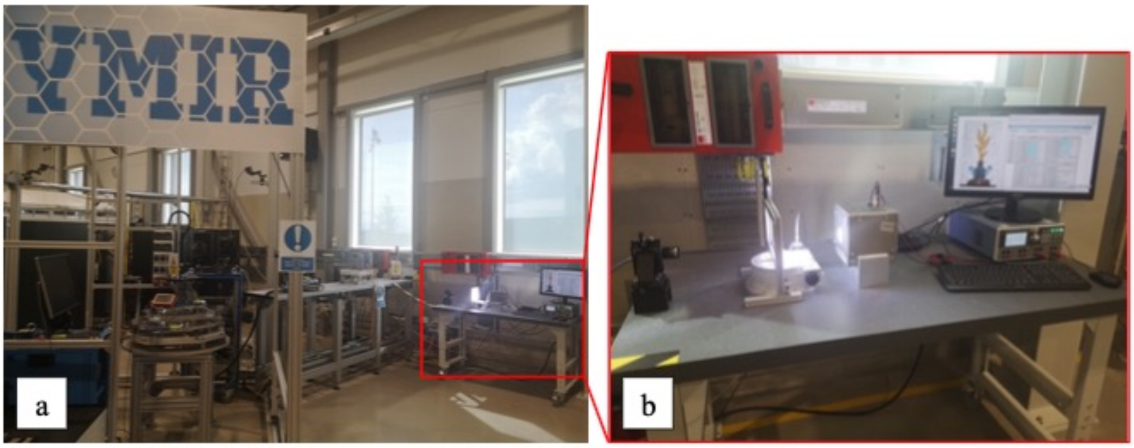


Figure 1: (a) The YMIR instrument at ESS; (b) the optical light tomography setup at YMIR

References

[1] https://nicos-controls.org
[2] https://epics.anl.gov
[3] https://kafka.apache.org
[4] https://www.nexusformat.org
[5] Larsson et al., 2022, IEEE Access: IEEE Education Society Section (submitted for publication)

Surfactant-free microemulsions : how molecular dynamic reflects nano-structuration

M K Firoz[a], Marie Plazanet[b], Ingo Hoffmann[b], Sylvain Prévost[b], Thomas Zemb[b] & Christiane Alba-Simionesco[b]

[a]PhD, Grenoble, France
[b]Researcher, Grenoble, France

malayil@ill.fr

Mixtures of simple molecules may lead to complex systems with unforeseen properties of interest such as reactivity or solubility. This is the case of aqueous solutions of alcohol and oil, typically found in all kind of liquors, cosmetics or solvents for liquid-liquid extraction.

The archetypical case of this family is the ternary mixture of water, ethanol and octanol. Its phase diagram presents a biphasic region with a critical point (Upper Solution Critical Temperature) close to which a strong nanostructuration of the liquid can be observed. In the biphasic region, this emulsion was termed Ouzo effect, following the Greek beverage, commonly observed when the 40 % alcohol liquor is quickly diluted with water. In the monophasic state, a « pre-Ouzo » region has aslo been evidenced [1], extending between identified frontiers around the critical point (see Fig. 1, [2]). The structuration is also characterized by a structure factor presenting an Ornstein-Zernike behaviour, indicating the presence of aggregates of the order of 100 molecules similar to a micro-emulsion formed by a ternary water-poor mixture of octanol and ethanol and water, surrounded by a surface excess of ethanol that is immersed in a binary water-ethanol solution saturated with a low quantity of octanol.

In this system, we investigated, using QENS and various isotopic mixtures, the relaxation dynamic of each component along different composition lines crossing the phase diagram. The evolution of the diffusion coefficient is measured over a wave vector ranging from ~0.05 A-1 to 0.6 A-1, bridging the scale from characteristic droplet size to molecular distances, i.e. from collective to individual dynamics. We will show how the dynamics also reflect the (nano)structural organisation.

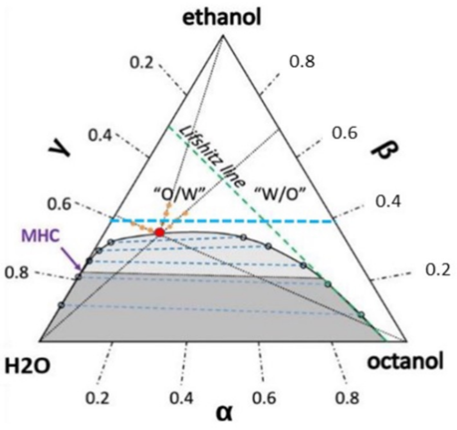


Fig. 1 : phase diagram (wt.%) of the ternary mixture octanol/ethanol/water. The binary region is colored in grey ; the Widom line starts from the critical point (red dot) and corsses the monophasic region toward the ethanol-water binary line ; the Lifshitz line delimitates the onset of structural organisation ; the minimum hydrotrope concentration (MHC) indicates the minimum quantity of ethanol required to form a monophasic solution as soon as octanol is added to water ; the dynamics was investigated along the blue dotted line.

References :

[1] T. Zemb et al., PNAS (2016) 113, 4260–4265

[2] Sylvain Prévost, Sebastian Krickl, Stjepan Marčelja, Werner Kunz, Thomas Zemb, and Isabelle Grillo, *Langmuir* **2021** *37* (13), 3817-3827.

Abstract: 179

Making neutron characterization tools available to industry

Hanna Leemreize[a], Nikolaj Zangenberg[a]
[a]Danish Technological Institute, Aarhus, Denmark
hle@dti.dk

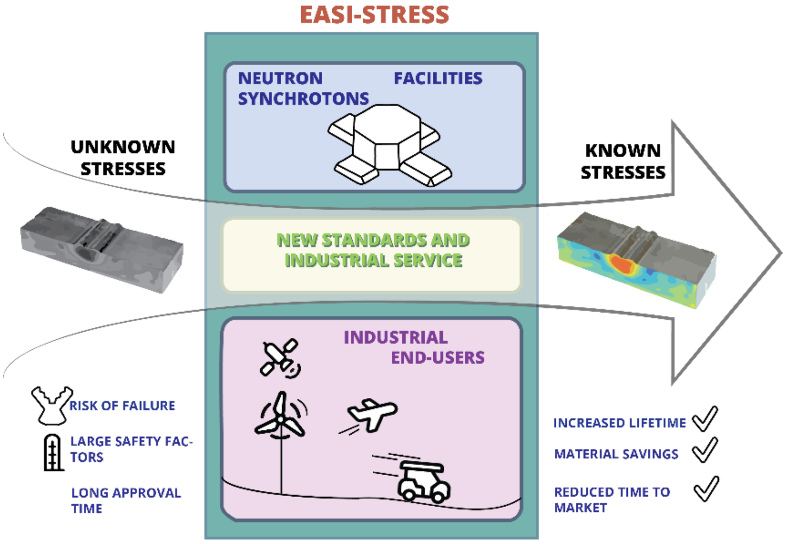
Large scale neutron facilities provide powerful tools to look into the interior of even large industrial components and get insights into materials and processes that would otherwise not be possible. Despite these unique capabilities, their industrial use remains limited.

An example of one such unique tool is measurement of bulk residual stresses by neutron diffraction. Although this characterization technique was developed in the 1980 and widely used in academia, no consensus has been reached in industry concerning how to use this powerful tool.

Being able to efficiently measure residual stresses will enable a better understanding of their formation and progression by direct comparison with and incorporation of the measured data into modelling tools. Incorporating this knowledge into the design process and lifetime assessment of metallic components will give more reliable products with increased lifetime and reduced material usage.

In the EASI-STRESS project [1], we aim to strengthen industrial access and uptake of non-destructive synchrotron and neutron diffraction-based residual stress characterisation tools by 1) validation against industrially accepted destructive tools and 2) development of harmonised protocols, and 3) introducing harmonised (meta)data formats and analysis software that are compatible with simulation tools.

With these results in-hand we can provide a service to industry ensuring traceability, reproducibility, and repeatability of the measured results.



References

[1] www.easi-stress.eu, European Union Horizon 2020 grant agreement No. 953219

First results with PASTIS3 at IN20

U. B. Hansen[a], B. Fåk[a], D. Jullien [a], A. Petroukov [a], M. Enderle [a]

[a]Institut Laue-Langevin, Grenoble, France

bengaardhansen@ill.fr

XYZ polarization analysis for inelastic neutron scattering is a powerful method for the separation of magnetic, nuclear coherent and incoherent scattering. However, in the thermal neutron range, these experiments have typically been carried out using the conventional triple-axis technique with a single analyzer and detector, where polarized cross sections are measured point by point. We will here present the results of the third generation PASTIS device for XYZ wide-angle polarization analysis [1]. The PASTIS3 device uses two independent polarized ^{3}He neutron spin filters for polarizing and analyzing the neutron spins. The incident neutron beam polarization can be inverted by reversing the spin state of the polarizing entrance cell and the ^{3}He analyzer cell provides a continuous 102-degree coverage of scattering angle. To obtain a high spatial homogeneity of the magnetic field in any field direction, a new coil design has been developed using sets of tilted coils. The setup has been tested on the thermal triple-axis instrument IN20 together with the multi-angle analyzer Flatcone.

References

[1] D. Jullien, A. Petoukhov, M. Enderle, N. Thiery, P. Mouveau, U. B. Hansen, P. Chevalier, P. Courtois, NIM A, **1010**, 165558 (2021).

A hitchhiker's guide to the easyScience galaxy

Simon Ward[a], Andrew Sazonov[a], Andrew R. McCluskey[a], Piotr Rozyczko[a]

[a]*European Spallation Source ERIC (Data Management and Software Centre)*

simon.ward@ess.eu

easyScience is an initiative from the European Spallation Source (ESS) to unify simulation software across neutron scattering. ***DON'T PANIC!*** While this goal seems an unsurmountable challenge, it is achievable as demonstrated by our current releases. The **easyScience** project has the following aims; provide a unified method to interact the most popular technique specific simulation software/libraries, a professional and welcoming graphical interface for new users, JuPyter notebooks for experienced users, unified data structures and workflows across multiple techniques.

As an opening to this project, Diffraction and Reflectometry techniques were chosen to demonstrate the *easy* philosophy. These techniques have multiple complex calculation engines available, which it is unrealistic to expect users to master. **easyReflectometry** and **easyDiffraction** unifies these calculation engines for their respective techniques and provides a complete, feature rich and easy to use interface. In the future QENS and spectroscopy will also be targeted. As a bonus, the technologies behind the **easyScience** programs allow for advanced modelling and statistical analysis techniques with the ability to scale for large datasets.

Behind these programs is **easyCore**, a unified simulation, optimization and analysis package. **easyCore** is built on the latest techniques and libraries including scipp (developed at ESS) for dataset handling, jax for machine learning and PyMC for Bayesian analysis. Hence all these features are available for all **easyScience** software. We present the main features of **easyScience**, where it came from, where it's going and how it will be used to enhance the analysis workflow with the latest analysis techniques.

Amorphous proteins in weakly hydrated environments: simulation vs. experiment

Mark Telling [a], Elisa Bassotti[1b], Gaio Paradossi [b], Sara Gabrielli [b] and Ester Chiessi [b]

[a] STFC, ISIS Facility, Oxfordshire, UK
[b] Dipartimento di Scienze e Tecnologie Chimiche, Università di Roma "Tor Vergata"

mark.telling@stfc.ac.uk

The characterization of biopolymers in the dry amorphous state has implications for the pharmaceutical industry since it provides deeper understanding regarding the effect of lyophilization on the stability and biological activity of bio-macromolecular drugs.

Using an in-silico approach, atomistic molecular dynamics simulation probe structural and dynamical features related to system functionality. However, atomistic simulation of lyophilized proteins is still a challenge since the available force fields and water models need to be carefully tested. There is also limited direct experimental information regarding protein-protein assembly to help guide design of the system to be simulated. Fortunately, the outputs from atomistic MD simulations align directly with those accessed by neutron scattering experiments in terms of the time and length scales probed. In particular, the method of Quasi-Elastic Neutron Scattering (QENS) can be used to investigate picosecond to nanosecond dynamics of molecular species and thus help validate MD protocols [1].

Here we report on Molecular Dynamics (MD) simulations of amorphous apoferritin and insulin, in both dry and low hydrated (h=0.31 for apoferritin and h=0.25 for insulin, h=g of D_2O/g of protein) conditions (T = 10 - 290 K). These proteins were chosen since they differ both in terms of complexity of the native biological assembly and secondary structure content. As such they can be used to better understand the influence of said factors on the local dynamics in the absence and presence of water molecules.

To perform these simulations, our team developed a novel in-silico lyophilization protocol which allowed us to build model dry systems directly from hydrated model starting structures. To test the results, we chose to monitor, as a dynamical observable, the temperature dependence of the mean squared displacement (MSD) of protein hydrogen atoms; a parameter accessible by both experimental QENS measurements and simulated neutron scattering spectra extracted from the MD trajectories. The agreement obtained between experiment [2] and simulation, in particular detection of the protein dynamical transition in the hydrated systems, validated the structural model descriptions used. Trajectory analysis also allowed us to follow the temperature evolution of secondary structures and thermal activation of individual protein residues and atomic groups.

References

[1] MTF Telling, A Practical Guide to Quasi-elastic Neutron Scattering, July 2020, Royal Society of Chemistry, ISBN: 978-1-78801-262-1. [2] Telling, MTF et al. "Mean squared displacement analysis of an-harmonic behavior in lyophilized proteins." Chem Phys 424 (2013): 32-36. doi:10.1016/j.chemphys.2013.05.008

Unique opportunities for particle physics at the ESS

Albert Young

North Carolina State University/Triangle Universities Nuclear Laboratory

aryoung@ncsu.edu

The European Spallation Source has incorporated into its baseline plan the capability to provide a special, very large beam port (incorporating the footprint for three typical neutron beams) and the possibility of second moderator below the "primary" moderator for condensed matter and materials studies. When combined with a facility layout which would permit the coupling of this port to an external experimental space or a very long (roughly 250 m) beamline, these features permit the design and operation of optimized experiments which are not practical at any other facility in the world. In fact, the high brightness of the upper moderator should also permit a new generation of beta decay and interferometry experiments which exploit the pulsed beam structure and very high brightness to push limits for short-ranged forces and explore aspects of phase-sensitive probes for new physics. In this presentation, I review some developing concepts to develop these opportunities for oscillations and interferometry experiments.

Henmilite: A quantum spin liquid candidate

Ankit Labh[a], *Dominik Legut*[a,b], *Petr Čermák*[a] *and Johanna K. Jochum*[c,d]

[a]*Charles University, Prague, Czech Republic*
[b]*IT4Innovations Center, VSB-Technical University of Ostrava, Ostrava, Czech Republic*
[c]*Maier-Leibnitz Zentrum (MLZ), Garching, Germany*
[d]*Technical University of Munich, Munich, Germany*

ankitlabh@gmail.com

Quantum materials have been playing a crucial role in the development of next-generation technologies and devices including quantum computers. Such materials are usually prepared under laboratory conditions. However some naturally occurring minerals, have also been found to feature complex magnetic ground states, such as Henmilite [1], Herbertsmithite [2]. They possess spin ½ Cu ions which exhibit a magnetic ground state favouring the creation of quantum fluctuations, hinting at a possible quantum spin liquid state [1].

Henmilite ($\mathrm{Ca_2Cu(OH)_4[B(OH)_4]_2}$) is only found in the Fuka mines of Japan [3], and has been suggested to consist of coupled two-leg ladders with an unusual antiferromagnetic dome [1] in the phase diagram pointing to the existence of quantum fluctuations making it a possible quantum spin liquid candidate. We plan to grow this material in the lab using isotopically pure starting materials, reducing its neutron absorption cross-section.

We will present first experimental results of the longitudinal thermal conductivity and the thermal Hall conductivity under an external magnetic field on Henmilite, together with the corresponding theoretical calculations. We are planning inelastic neutron scattering experiments using three axes, time-of-flight and/ or MIEZE spectroscopy to capture the dispersion of magnons in the vicinity of the ordered state. We will use the newly developed instrument ALSA [4],an AI-controlled robotic arm to co-align smaller crystals into mosaics to use in neutron spectroscopy.

References

[1] Hajime et al, PRM 5, 104405 (2021)
[2] Braithwaite et al, Mineralogical magazine 68.3 (2004): 527-539.
[3] Izumi Nakai et al, American Mineralogist 1986; 71 (9-10): 1234–1236.
[4] https://mambaproject.cz/alsa

Abstract: 187

Human transthyretin and amyloidosis

Mizar F. Oliva[1,2,3], Alexander F. Minns[1,3,4], Clare L. De'Ath[1,3,4], Matthew P. Blakeley[1], Valentina Mondani[5], Martine Moulin[1,3], Daniel Sarabi[9], Christoph Mueller-Dieckmann[3,4], Matthew W. Bowler[4,6], Edward P. Mitchell[2,3,4], Michael Haertlein[1,3],Vittorio Bellotti[5,7] and V. Trevor Forsyth[1,2,3,8,9]

[1]Institut Laue-Langevin, 71 avenue des Martyrs, Grenoble 38042 Cedex 9, France.
[2]Faculty of Natural Sciences, Keele University, Staffordshire ST5 5BG, U.K.
[3]Partnership for Structural Biology, 71 avenue des Martyrs, Grenoble 38042 Cedex 9, France.
[4]European Synchrotron Research Facility, 71 avenue des Martyrs, Grenoble 38042 Cedex 9, France
[5]Department of Molecular Medicine, Institute of Biochemistry, University of Pavia, 27100 Pavia, Italy
[6]EMBL Outstation, 71 avenue des Martyrs, Grenoble 38042 Cedex 9, France.
[7]Wolfson Drug Discovery Unit, Centre for Amyloidosis and Acute Phase Proteins, Division of Medicine, University College London, London NW3 2PF, UK
[8]Faculty of Medicine, Lund University, SE-221 84, Lund, Sweden
[9]LINXS Institute of Advanced Neutron and X-ray Science, Scheelevagen 19, 223 70 Lund, Sweden

Human Transthyretin (TTR) is a plasma protein implicated in several forms of amyloidosis. Many TTR point mutations have been characterized, partially elucidating the mechanisms that underlie the fibrillation process *in vivo*. Here we describe detailed structural and biophysical studies of a novel and highly pathological variant of TTR in which two residues are inserted at the level of the CD loop. This variant, which was identified clinically, imparts unusually aggressive amyloidosis as characterised *in vivo* and during *ex vivo* studies using material derived from the afflicted patient. A full crystallographic study of this protein has been carried out using both neutron and X-ray crystallography as well as biophysical characterization of its instability as a function of pH, guanidine thiocyanate and temperature alongside with fibril formation *in vitro*. The results provide clear evidence that this TTR variant is substantially less stable than the most pathological mutants that have so far been described. The structural studies point to the CD loop of the protein as the major factor underlying the protein instability and to the aggressiveness of this TTR mutant *in vivo*. Complementing molecular dynamics studies are being carried out on this system.

The upgrade of the thermal neutron TAS at ILL: from IN8 to THERMES

Andrea Piovano,[a] *Alexander Ivanov*[a]

[a] *Institut Laue-Langevin (ILL), 38042 Grenoble, France*

piovano@ill.fr

The three-axis spectrometer IN8 provide to ILL users an extremely high neutron flux and advanced conditions for studies of thermal excitations in single crystals and liquids. The use of, several and exchangeable, large double-focusing monochromators and analysers providing high counting rate even for small and low-scattering samples ensure the instrument performance and flexibility.

The new monochromator unit has been recently commissioned and profit of the fruit of the experience accumulated in the former TAS-group at ILL in using Bragg-focusing beam optics with independently variable and remotely controlled horizontal and vertical focusing (bending) of the crystal reflecting planes. The new monochromator for the thermal neutron beam considerably outperforms the previously used one. It consists of 4 different exchangeable crystal planes. The two planes are built from mosaic crystals of pyrolytic graphite and copper with the principal reflections PG002 and Cu200 chosen to provide a broad range of monochromatic neutron wave vectors and energy resolution of the incident beam. The other two planes are assembled with elastically bent perfect silicon crystals set to make use of the reflections Si111 and Si311 with prohibited second-order diffraction harmonics. The mosaic crystal planes are used in experiments requesting maximum monochromatic intensity at the sample position and variable resolution. The silicon crystal planes, with similar to mosaic crystals available resolution range, provide particularly "clean" conditions for experiments with multi-analyser configurations, such as the routinely used FlatCone, at the expense of marginally lower monochromatic flux.

The further step in renovation of the spectrometer is a classical single-detector secondary spectrometer setup called THERMES (THERMal Excitations Spectrometer) now commissioned at IN8. The new instrument benefits from a compact design that permits a larger accessible dynamic range (wider available angular ranges in the existing experimental zone) with particular attention paid to neutron shielding including special construction of the detector diaphragm. The user experiments with THERMES have been routinely performed over the last few reactor cycles showing an enhanced signal to noise ratio.

Further development of specific sample environment for this spectrometer is under way.

Foam aging under free drainage analysed using associated operando techniques

MJulien Lamolinairie[a], Benjamin Dollet[b], Jean-Luc Bridot[c], Olivier Diat[d] and Leonardo Chiappisi[a]

[a]ILL, Grenoble, France
[b]LIPhy (CNRS), Grenoble, France
[c]Teclis Scientific, Civrieux-d'Azergues, France
[d]ICSM (CEA/CNRS), Marcoule, France.

lamolinairie@ill.fr

Liquid foams are non-equilibrium multi-scale soft structures for which stability is still difficult to predict. In order to better understand the physico-chemical factors that affect the aging of foams at different length scales, a new columnar device was developed. It enables the simultaneous data collection from a small-angle neutron scattering (SANS) diffractometer, from an optical camera and an electrical conductivity meter. In order to evaluate the potentiality of this device, a foam from a mixture of the nonionic alkylether BrijO10 surfactant and of the sodium dodecyl sulfate (SDS) anionic surfactant was generated in D2O and analyzed as a function of time.

The volume of the foam, its liquid fraction and the radius of the foam bubbles as a function of time (obtained by image analysis and conductivity processing) are macroscopic information, necessary for foam lifetime characterization but not sufficient. SANS analysis is known to provide useful information at the nano-scale, on the structure of the inter-bubble film or the surfactant self-assemblies trapped within the foam [1-3]. However, to extract this information over a large structural range and in order to be able to compare the various structural features (the specific surface area, the liquid fraction, the film thickness) determined from different techniques, we have for the first time performed a quantitative analysis - in absolute scale - of the scattering data.

The analysis of nano- and macroscopic information obtained simultaneously enabled us to better understand the correlation between the mechanisms of drainage, ripening and coalescence involved at the different scales in the aging of the foam.

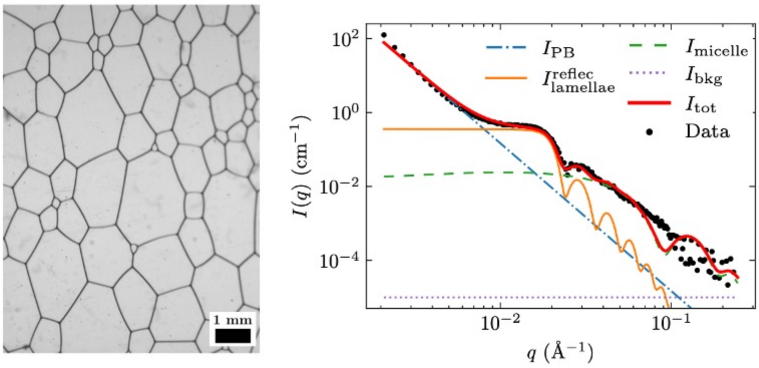


Figure 1: Left - Picture of a foam obtained for a 65 min aging time (free drainage mode). Right - Various contributions of the total scattering simulation (red curve) to adjust SANS data from a foam as photographed in left picture : I_{PB} *from Plateau Borders,* $I_{micelle}$ *from micelles present within the foam,* $I^{reflec}_{lamellae}$ *from lamellae.*

References :

[1] M. Axelos, et al, Langmuir, 19, 6589-6604, 2003
[2] M. Hohenschutz, et al, JCIS, 603, 141-147, 2021 ,
[3] A Mikhailovskaya, et al, Advances in Colloid and Interface Science, 247, 444-453, 2017