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Kiel

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Editorial

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Deutsche Neutronenstreutagung 2016,
Christian-Albrechts-Universität zu Kiel
September 2016

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Christian-Albrechts-Universität zu Kiel


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Foreword

The German Conference on Neutron Scattering 2016 - Deutsche Neutronenstreutagung 2016 (Kiel, September 20-22, 2016) - offers a forum for the presentation and critical discussion of recent results obtained with neutron scattering and complementary techniques. It is organized on behalf of the German Committee for Research with Neutrons - Komitee Forschung mit Neutronen, KFN - by the Helmholtz-Zentrum Geesthacht and the Christian-Albrechts-Universität zu Kiel. After the conference, a workshop „Demands of European neutron users in materials science for the instrumentation of the upcoming PIK neutron science centre“ will take place in the frame of the EU project „CREMLIN - Connecting Russian and European Measures for Large-Scale Research Infrastructures“ (Kiel, September 22-23, 2016).

The conference offers the vibrant German and international neutron community an opportunity to debate topical issues in a stimulating atmosphere. Originating from “BMBF Verbundtreffen” - meetings for projects funded by the German Federal Ministry of Education and Research - this conference series has a strong tradition of providing a forum for the discussion of collaborative research projects and future developments in the field of research with neutrons in general, which provides a unique chance for exploring interdisciplinary research opportunities. It also serves as a showcase for recent method and instrument developments and to inform users of new advances at neutron facilities. This year, minisymposia will focus on the topics „Proteins: dynamics and interactions“, „High brilliance compact neutron sources - status and perspectives“, „Neutron spectroscopy for dynamics in confinement“ and „Materials characterisation“.

Welcome to Kiel!
General Information

Venue
General Information

Conference Office
The conference office (dn2016@sni-portal.de) is located in the Audimax and is open during these periods:

- Tuesday, September 20: 08:00 - 20:00
- Wednesday, September 21: 08:30 - 18:15
- Thursday, September 22: 08:30 - 14:00

Lunch
Participants receive meal vouchers at registration. These can be used for lunch in the nearby Mensa I. There is a separate dining room reserved for the conference upstairs. On Thursday, we will offer a packed lunch at the conference venue.

Presentations
There are projectors and computers installed in the lecture halls, but you can also use your own laptop. Please prepare in time for your presentation. Poster mounting material is available at the conference office.

Evening lecture
On Tuesday, September 20, Prof. Stephan Paul will hold an evening lecture on „Das Neutron und das Universum“. This lecture is open to the general public.
Conference Dinner
On the evening of Sept. 21, 2016, the conference participants are invited for a round cruise on the Kieler Förde aboard the „Adler Princess“. Bus transfer to Sartorikai will start at 18:15 in front of the Audimax. Boarding starts at 18:45, departure is scheduled for 19:00. The ship will return to Sartorikai around 22:00. Since most hotels are located in town, the return has to be organised individually. Closest bus stops are Eggerstedtstraße, Schwedenkai, Seegarten Ostseekai, Bootshafen and Holstenbrücke.
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The European Spallation Source: From Construction to Science

Andreas Schreyer
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Keywords: neutron facilities, instrumentation

After a short summary of the design and the specifications of the ESS an overview of the current status and schedule of the ESS construction project will be given with a strong focus on the instruments and the surrounding scientific infrastructure. The overall goal of ESS is to begin user operation with 8 instruments in 2023. The plan is to reach the full scope of 22 public instruments by 2028. ESS will cover a very broad spectrum of science and is designed to push the limits of neutron methods to new horizons. Selected examples of new scientific opportunities will be discussed.
Materials Sciences  

Keynote lecture, Tuesday 09:50-10:15

3D lithium distribution in Li-ion batteries revealed in situ by neutron scattering

Anatoliy Senyshyn
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Keywords: neutron, Li-ion battery, structure, homogeneity

Energy storage media based on different technologies have gained in importance for a wide field of applications ranging from supplying portable devices to large electric vehicles. In recent decades the energy storage technology based on lithium ions is dominating the marked due to its 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 new dedicated experimental techniques capable to reveal “live” information about processes occurring inside the cell. In such instance neutron scattering is already a well-established experimental technique for the characterization of Li-ion batteries. Neutron scattering methods when combined with electrochemical characterization undergo an increasing relevance for studies of lithium-ion based electrochemical energy storage systems on different length scales, e.g. neutron imaging, reflectometry, small-angle neutron scattering, quasielastic neutron scattering and powder diffraction. In-situ experiments with neutrons are performed on self-developed/special test cells and commercial Li-ion cells of different designs depending on the research needs.

Simple in principle but complicated in practice designs of modern Li-ion batteries may result in spatial inhomogeneity of current, lithium or electrolyte distribution, which are often difficult to quantify, but it will surely affect performance, cycling stability and safety. Despite the increasing popularity of neutron scattering studies of batteries at their operating conditions the problem of cell homogeneity (indirectly pointed by electrochemical measurements) and its effect on the data is often not properly accounted in literature. Here a combination of three neutron-based experimental techniques, namely computed neutron tomography, high-resolution neutron powder diffraction and spatially-resolved neutron powder diffraction, applied in situ for studies on commercial Li-ion cells of the 18650-type will be presented along with results of electrochemical studies. The details of the cell organization on different length scales and its evolution on various factors like state of charge, temperature and fatigue will be discussed in light of 3D lithium distribution in cylinder-type Li-ion batteries.

References:
Session 1: Materials Sciences

Tuesday, Sept. 20, 2016, 10:45 - 12:15. Session Chair: Andreas Schreyer
Location: Frederik-Paulsen-Hörsaal

10:45 – 11:00: **Diffusion Studies in Amorphous Si and Si-Li Compounds Using Neutron Reflectometry (ID 148)** Florian Strauß, Erwin Hüger, Jochen Stahn, Thomas Geue, Stefan Mattauch, Alexandros Koutsioubas, Harald Schmidt

11:00 – 11:15: **In-Situ Polarised Neutron Reflectometry During Thin Film Growth (ID 112)** Wolfgang Kreuzpaintner, Sina Mayr, Jingfan Ye, Birgit Wiedemann, Amitesh Paul, Thomas Mairoser, Andreas Schmehl, Alexander Herrnberger, Jochen Stahn, Jean-Francois Moulin, Panagiotis Korelis, Martin Haese, Matthias Pomm, Björgvin Hjörvarsson, Peter Böni, Jochen Mannhart


11:30 – 11:45: **Ordering and Disordering of β Phase in TiAl Alloys in Dependence of Alloy Composition (ID 116)** Victoria Kononikhina, Andreas Stark, Florian Pyczak, Weimin Gan, Andreas Schreyer

11:45 – 12:00: **In-situ Studies of Thin-Film Composite Reverse Osmosis Membranes With SANS during the Process of Wastewater Desalination (ID 177)** Dietmar Schwahn, Roni Kasher, Vitaliy Pipich, Yoram Oren

12:00 – 12:15: **Transformation of CTAB Bilayers into Micelles by Desorption from Gold Nanorod Surfaces – A combined SAXS/SANS study (ID 202)** Tilo Schmutzler, Torben Schindler, Peter Lindner, Tobias Unruh
Diffusion Studies in Amorphous Si and Si-Li Compounds Using Neutron Reflectometry

Florian Strauß, Erwin Hüger, Jochen Stahn, Thomas Geue, Stefan Mattauch, Alexandros Koutsioubas, Harald Schmidt

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Keywords: neutron reflectometry, dynamics, diffusion, energy materials

Neutron reflectometry (NR) is a powerful tool in diffusion studies due to its ability to determine diffusion lengths of 1 nm and below. In recent years the technique has been employed on suitable multilayer samples of metastable materials. As a first example amorphous silicon (a-Si) was investigated. a-Si is found in solar cells, display technologies and is under investigation as a promising electrode material in Li-ion batteries. For the study of self-diffusion in a-Si \(^{29}\text{Si}/^{28}\text{Si}\) multilayer samples were deposited by ion beam sputtering and subsequently annealed in Ar atmosphere. Diffusivities following the Arrhenius law were determined in the temperature range between 550 and 700 °C.\(^1\) At lower temperatures between 400 and 500 °C a time-dependent short range diffusion process is found and interpreted as a result of structural relaxation.\(^2\) Additionally, since for the lithiation of a-Si electrodes in Li-ion batteries amorphous Li,Si phases play an important role, atomic mobility of Li through Li,Si layers was probed. Li,Si layers were produced by co-sputtering of a segmented Li-Si target.\(^3\) For NR measurements such layers were deposited as intermediate layers between alternating \(^6\text{LiNbO}_3\) and \(^{nat}\text{LiNbO}_3\) layers in a multilayer stack. Transport parameters were determined\(^4\) using a method recently proposed by Hüger, et al.\(^5\) Additional measurements by Secondary Ion Mass Spectrometry confirm the data obtained by NR.

References:
In-Situ Polarised Neutron Reflectometry During Thin Film Growth

Wolfgang Kreuzpaintner¹, Sina Mayr¹, Jingfan Ye¹, Birgit Wiedemann¹, Amitesh Paul¹, Thomas Mairoser², Andreas Schmehl², Alexander Hernnberger², Jochen Stahn⁵, Jean-Francois Moulin⁴, Panagiotis Korelis⁵, Martin Häse⁴, Matthias Pomm⁴, Björgvin Hjörvarsson⁶, Peter Böni¹, and Jochen Mannhart³

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² Universität Augsburg, Augsburg, Germany
³ Max-Planck-Institut für Festkörperforschung, Stuttgart, Germany
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⁵ Paul Scherrer Institut, Laboratory for Neutron Scattering, Villigen PSI, Switzerland
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Keywords: in-situ, polarised, neutron, reflectometry, thin film, growth

Thin magnetic layers and heterostructures thereof are the basic building blocks of a large number of magneto-electronic devices. Their performance strongly relies on the magnetic properties of the layers they consist of. These are functions of the layers' morphology and microstructure and on the coupling between them. Since these parameters can change during the process of growth, it is important for the understanding and optimisation of magneto-electronic devices to not only accurately monitor the structural but also the magnetic properties during the process of growth.

While the structural characterisation of thin films during growth by various techniques is common practice (as e.g. commonly done by RHEED/LEED, STM or synchrotron radiation), the in-situ measurement of the magnetic properties of films using (polarised) neutron reflectometry is a challenging task. Within a collaboration of TU München, University Augsburg and MPI Stuttgart, we operate a mobile sputtering facility for the growth and in-situ monitoring of magnetic multilayers, which can be installed at suitable neutron beamlines. In our contribution, the setup and first proof of principle polarised in-situ neutron reflectivity measurements on in-situ grown Fe/Cr carried out at the ToF reflectometer REFSANS at the FRM II neutron source and at the AMOR beamline at PSI will be presented. At the latter, use of the Selene neutron optical concept allows very fast polarised neutron reflectivity measurements to be performed within only 15min per spin direction.
Local structure and proton transport in HT-PEFCs measured with neutron scattering

Olaf Holderer, Oxana Ivanova, Marina Khaneft, Bernhard Hopfenmüller, Wiebke Lüke, Anne Majerus, Marie-Sousai Appavou, Noemi Szekely, Margarita Krutyeva, Reiner Zorn, and Werner Lehnert

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Keywords: neutron, instrumentation, dynamics, soft matter

The conception, design and optimization of fuel cells in general requires a good knowledge of the underlying physical processes on all length scales. In this contribution we focus on High Temperature Polymer Electrolyte Fuel Cells (HT-PEFCs) based on a proton conducting membrane such as poly2,2’-m-(phenylene)-5,5’-bibenzimidazole (PBI) doped with phosphoric acid (PA) and show how microscopic neutron scattering techniques can be related to macroscopic properties of the fuel cell. Contrast variation by using different isotopes and the sensitivity to light elements are the major advantages of neutron scattering. Small angle neutron scattering (SANS) provides insight into the fractal structure of the PA doped membrane on length scales of ~10-500 nm. With quasielastic neutron scattering (QENS), proton diffusion can be measured on local length scales of about 0.1-10 nm [1,2]. The length scale dependent energy transfer measured with QENS gives insight into local proton transport processes in the PBI membrane and in the adjacent electrode layers and allows to relate microscopic proton mobilities with macroscopic measurements, e.g. of the proton conductivity of the membrane [3]. Complementary techniques such as X-ray scattering, TEM or PFG-NMR round up the picture on a broad range of length scales.

Ordering and Disordering of $\beta$ Phase in TiAl Alloys in Dependence of Alloy Composition

Victoria Kononikhina$^1$, Andreas Stark$^1$, Florian Pyczak$^1$, Weimin Gan$^2$, Andreas Schreyer$^3$

$^1$ Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Geesthacht 21502, Germany;
$^2$ Structural Research on New Materials, HZG Outstation at FRM II, Garching, Germany;
$^3$ European Spallation Source ERIC, Lund, Sweden;
E-Mail: Victoria.kononikhina@hzg.de;
Keywords: Ti-Al alloys, phase transformation, neutron diffraction, SEM

Due to their low density (4 g per cm$^3$), good oxidation and corrosion resistance and high specific tensile and creep strength, $\gamma$-TiAl based alloys recently have started to replace Ni-based superalloys as a material for turbine blades in aircraft engines. TiAl alloys in the range of 39 – 45 at.% Al usually contain the ordered phases $\gamma$ and $\alpha_2$ at lower and disordered $\alpha$ at higher temperatures. Depending on alloy composition and temperature additionally disordered $\beta$-Ti(Al) (A2 structure) or ordered $\beta_o$-TiAl (B2 structure) can occur. The ductile high temperature $\beta$ phase is important for the processing of the material while the low temperature $\beta_o$ phase is said to embrittle the material at service temperature.

We used the good contrast of neutron diffraction for ordered and disordered $\beta$ phase of TiAl-based alloys to determine the order/disorder temperatures which are not accessible by other methods like DSC measurements. Three binary TiAl alloys (Ti-xAl with x = 39, 42 and 45) and five alloys with additional alloying elements (Ti-42Al-2Y with Y = Nb, Mo, Ta, Cr and Fe) were used to investigate the influence of different Al concentrations and alloying additions on the kinetics of the occurring ordering/disordering reactions and phase transformations. In the presentation new results of neutron diffraction experiments and microstructure investigations by scanning electron microscopy will be shown.
In-situ Studies Of Thin-Film Composite Reverse Osmosis Membranes With SANS During The Process Of Wastewater Desalination

Dietmar Schwahn 1, Roni Kasher 2, Vitaliy Pipich 3, Yoram Oren 2

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2 Zuckerberg Institute for Water Research, Jacob Blaustein Institute for Desert Research, Ben-Gurion University of the Negev, Sede Boqer Campus 84990, Israel.
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Keywords: small-angle neutron scattering, biomineralization, reverse osmosis wastewater desalination

Formation of aggregates caused by the interaction of organic and inorganic molecules, in particular those involved in the formation of calcium phosphate minerals, have strong negative influence on membrane permeability in reverse osmosis (RO) wastewater desalination. Presently, RO is an important tool for gaining and recovering potable water. A better understanding of the origin of these aggregates is urgently needed for economic reason but also for scientific purpose as these phenomena have very much in common with the broad and versatile field of biomineralization. Small-angle neutron scattering (SANS) is a strong tool in this field as it is allowing quantitative analysis on microscopic length scale and differentiates between organic and inorganic components. One of our goals is the realization of in-operando SANS experiments on RO wastewater desalination under most realistic conditions. For this purpose we developed a cell for real-time SANS experiments simulating the process of reverse osmosis (RO) wastewater desalination up to 30 bar as is shown in the Figure. These studies are quite complex from the point of experimental set-up and from the interpretation of the scattering itself. The scattering has to be differentiated from three different sources, namely from feed, fouling layer at the surface of the membrane, and from the membrane itself. It is shown that the scattering from RO membranes is very strong and dominating. In parallel we always measured the permeability of the membrane which is an important parameter for the engineer.

In my presentation I will describe the in-operando cell as well as present SANS data from the various topics of this project. These are: (i) Formation and characterization of aggregates formed in a simulated secondary effluent (SSE) after adding various organic molecules, (ii) characterization of RO thin-film composite (TFC) membranes using SANS contrast variation, and (iii) first results from desalination experiments performed at the very-small angle scattering (VSANS) instrument KWS3 of the FRM II.

Transformation of CTAB Bilayers into Micelles by Desorption from Gold Nanorod Surfaces – A combined SAXS/SANS study

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²Institut Laue-Langevin, F-38042 Grenoble, France
Keywords: SAXS, SANS, gold nanoparticles, CTAB, micelles, stabilization, adsorption, desorption

The plasmonic behaviour of gold nanorods is strongly dependent on their size and shape. Their synthesis includes high amounts of the structure directing agent and micelle forming surfactant hexadecyl-trimethyl-ammonium bromide (CTAB).[1] One common route of producing AuNRs is to reduce the precursor HAuCl₄ in the presence of small seed particles, CTAB and AgNO₃. Although the synthesis of AuNRs is highly investigated the exact role of CTAB and AgNO₃ is still unclear.[2] CTAB forms a 32 Å thick stabilizing bilayer.[3] AgNO₃ stays unreduced after the reaction whereas the concentration is crucial regarding the final morphology of the gold NPs. The common explanation for that effect deals with the adsorption of silver complexes beside CTAB on certain facets of the gold nanocrystals but a clear proof is still missing. We studied the adsorption of silver complexes (and the desorption of CTAB) by a combination of small angle scattering (SAS) with X-rays (SAXS) and neutrons (SANS) and transmission electron microscopy (TEM).

Fig.1: TEM image (left) of a sample prepared of the AuNR dispersion used for further SAS analysis. Fitting the SAS data (of SANS @ D11, ILL in Grenoble, France and SAXS @ our laboratory instrument) simultaneously is very sensitive to the rod diameter distribution and can be compared directly to the values gathered from quantitative TEM analysis (not shown). The addition of silver and thiourea (tu) as ligand leads to a strong modification of the SANS signal (right). The scattering can be explained by the formation of CTAB micelles. This is consistent with an increased electron density of the AuNR shell (analysed by SAXS) and can be explained via the partial transformation of the CTAB bilayer on top of the AuNRs into micelles due to the replacement of the molecules by the much stronger adsorbing silver-thiourea complexes.

Silver-thiourea complexes seem to adsorb much stronger on the AuNR surfaces compared to the CTAB molecules. This leads to a partial transition of the CTAB stabilizing layer into micelles which becomes visible due to the formation of a strong scattering in SANS that is characteristic for dispersed CTAB micelles in aqueous solution (figure 1, right). As the concentration of the silver complex molecules at the gold nanorods facets increases, the electron density of the nanorod shell is significantly enhanced, which was detected by fitting the SAXS data of the different solutions. Using the morphologic distribution (analysed by TEM) and the shell thickness of 32 Å determined by SAXS and SANS[3] it was possible to fit the SAS data simultaneously to analyse the electron density of the nanorod shell and the amount of released micelles.

References:
[2] F. Hubert, F. Testard, O. Spalla; Cetyltrimethylammonium Bromide Silver Bromide Complex as the capping Agent of Gold Nanorods; Langmuir, 24, 9219 (2008); DOI: 10.1021/la801711q
M1_1: Minisymposium
“Proteins: Dynamics and Interactions”, part 1

Tuesday, Sept. 20, 2016, 10:45 – 12:15. Session Chair: Roland Winter
Location: Hörsaal C

10:45 – 10:55: Welcome by Organizers, Frank Schreiber

10:55 – 11:20: Bridging Attraction Colloidal System: a Model System for Some Ion Specific Effects in Protein Solution (ID 275) Yun Liu

11:20 – 11:45: Dynamics of Crowded Protein Solutions (ID 276) Anna Stradner

11:45 – 12:00: Fast Antibody Fragment Motion: Flexible Linkers Act as Entropic Spring (ID 114) Ralf Biehl, Laura Stingaciu, Oxana Ivanova, Dieter Richter

12:00 – 12:15: Animated Symposium Discussion Session, Roland Winter
Bridging attraction colloidal system: a model system for some ion specific effects in protein solution

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Keywords: neutron, colloid, protein, interaction, bridging attraction

Abstract: Understanding protein-protein interaction is the key to control its phase behavior and macroscopic solution properties for many applications such as protein crystallization and protein formulations of cancer treatment drugs. One of the mysterious behavior of protein-protein interactions is the ion specific effect, which cannot be explained by the conventional Debye-Hückel and DLVO theory where ions are treated as point charges. One famous example is the Hofmeister series where different types of ions have different capability in sorting out or sorting in proteins. Different theories have been proposed to understand these ion specific effects. Here, we show our theoretical results together with the experimental observations that the size of small particles, such as ions, and their interactions with large colloidal particles, such as proteins, are important for its phase behavior.$^{1,2}$ At the extreme case of the strong attraction between small and large particles, the liquid reentrance effect is predicted as small particles can introduce the bridging attraction that is significantly different from the widely studied depletion attraction.$^1$ We have tested our theoretical prediction experimentally with a colloidal system mixed with small and large particles whose size ratio is comparable to that between ions and proteins. We observed a liquid-gel-liquid transition experimentally consistent with our theoretical prediction.$^2$ Our results are also consistent with the reentrance phase diagram observed in BSA protein solutions with trivalent counterions.

References:
Dynamics of Crowded Protein Solutions

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Keywords: neutron spin echo, interactions, patchiness

Many biopharmaceuticals such as proteins or peptides require high concentrations to allow for injecting effective doses in a single dose delivery. This in turn poses considerable challenges for the formulation, as under these crowded conditions the stability and the dynamic properties such as the resulting viscosity are very difficult to predict and control. It is thus essential to achieve a quantitative understanding of the link between the molecular structure of the proteins and the interactions between them, and how these interactions influence the stability, dynamics and flow properties of the solutions as a function of their concentration. Here we show how we can use a combination of advanced characterization techniques\(^1\) such as neutron spin echo (NSE), small-angle scattering and 3D cross correlation light scattering (3DDLS), combined with state-of-the-art computer simulations to assess and predict interparticle interactions and their impact on the dynamics and flow behavior of crowded protein solutions. We particularly point out the enormous influence of weak attractive interactions known to exist between many globular proteins, and demonstrate the dramatic effect of an interaction potential anisotropy such as attractive patches on the dynamic properties.\(^2\)

References:
Fast Antibody Fragment Motion: Flexible Linkers Act as Entropic Spring

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Keywords: neutron, protein dynamics, antibody, entropic spring

A flexible linker region between three fragments allows antibodies to adjust their binding sites to an antigen or receptor. Using Neutron Spin Echo Spectroscopy we observed fragment motion on a timescale of 7 ns with motional amplitudes of about 1 nm relative to each other.¹ The mechanistic complexity of the linker region can be described by a spring model with Brownian motion of the fragments in a harmonic potential. Displacements, timescale, friction and force constant of the underlying dynamics are accessed. The force constant exhibits a similar strength to an entropic spring, with friction of the fragment matching the unbound state. The observed fast motions are fluctuations in pre-existing equilibrium configurations. The Brownian motion of domains in a harmonic potential is the appropriate model to examine functional hinge motions dependent on the structural topology and highlights the role of internal forces and friction to function. We compare the results to internal motions found for other proteins with different topology.

References:
M2_1: Minisymposium  
“High brilliance compact neutron sources – status and perspectives”, part 1

Tuesday, Sept. 20, 2016, 10:45 – 12:15. Session Chair: Thomas Gutberlet  
Location: Hörsaal D

10:45 – 11:30: The HBS Project: A High Brilliance Neutron Source (ID 264)  
Thomas Brückel, Ulrich Rücker, Tobias Cronert, Paul Zakalek, Thomas Gutberlet, Jörg Voigt, Yannik Beßler, Michael Butzek, Jan Philipp Dabruck, Paul-Emmanuel Doege, Rahim Nabbi, Carsten Lange, Marcel Klaus

11:30 – 11:50: RIKEN Accelerator-driven Compact Neutron Source, RANS, and its Practical Applications (ID 263)  
Yoshie Otake

Richard Dronskowski
The HBS Project: A High Brilliance Neutron Source

Thomas Brückel¹, Ulrich Rücker¹, Tobias Cronert¹, Paul Zakalek¹, Thomas Gutberlet¹, Jörg Voigt¹, Yannik Beßler², Michael Butzek², Jan Philipp Dabruck¹, Paul-Emmanuel Doege³, Rahim Nabbi³, Carsten Lange⁴, Marcel Klaus⁵

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Keywords: neutron source development

With the construction of the ESS, the European neutron user community is looking forward to the brightest source worldwide. At the same time there is an ongoing concentration of research with neutrons to only a few but very powerful neutron facilities. Responding to this situation the Jülich Centre for Neutron Sciences has initiated a project for a compact accelerator driven high brilliance neutron source, optimized for neutron scattering on small samples and being realized at reasonable costs. The project deals with the optimization of potential projectiles and target to moderator concepts, versatile accelerator systems, cold sources, beam extraction systems and optimized instrumentation. A brief outline of the project and reached achievements will be presented¹ as well as a vision for the future neutron landscape in Europe.

References:
RIKEN Accelerator-driven Compact Neutron Source, RANS, and its Practical Applications

Yoshie Otake
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RIKEN accelerator-driven compact neutron source (RANS) has been developed and provided neutrons for industrial use. The proton linac of 7MeV with the maximum average current 100 μA, pulse width 10us-180us, repetition frequency 20-200Hz is used with long-life Be target for such practical use in the field of manufacturing. The success of the visualization of the corrosion and the water movement with wet-dry process under the film in the normal steel and corrosion –resistant alloy steel samples with RANS has clarified high potential of compact neutron source for metal and steel samples. The texture evolution due to plastic deformation was successfully observed by measuring a change in the diffraction peak intensity using TOF measurements with 2D detector, furthermore, the volume fraction of the austenitic phase in the dual phase mock specimen was also successfully evaluated by fitting the diffraction pattern using a Rietveld code. Another important mission of our project is to develop novel non-destructive inspection system for large scale infrastructures such as pre-stress concrete bridges with transportable compact neutron source including high sensitive detector system. The difference of the number of the steel bars in the thick concrete slab successfully distinguished with the new pixel fast neutron imaging detector, the air gap and the water under the thick concrete block are also measured. For more compact neutron source, we plan to construct RANS2 with 2.49MeV proton linac. The further plan will be discussed in the presentation.
Solid State Chemistry and Neutron Powder Diffraction

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Keywords: chemistry, nitrogen, neutron diffraction, POWTEX

Since the beginning of the digital age, one is puzzled to note an ever-increasing pressure on conference speakers forcing them to deliver scientific abstracts through Web-based interfaces months ahead of the actual conferences. Not only does this nuisance increase the frustration of the speakers (who typically suffer from other unnecessary burdens of digital bureaucracy such as Excel sheets), it furthermore leads to a lack of spontaneity, less timeliness and, thus, an increasing number of duller talks. In my presentation, I will cover my usual mixture between advanced solid-state chemistry and neutron diffraction, and I will include the most newsworthy results from my group according to my honest judgment. The results will be most probably inspired by the realm of complex nitrogen-based materials such as nitrides, carbodiimides, and guanidinates, and they will also strongly touch upon the high-performance time-of-flight neutron diffractometer POWTEX presently under construction at the FRM II in Garching.
Session 2: Condensed Matter Research

Tuesday, Sept. 20, 2016, 13:45 - 15:15. Session Chair: Wiebke Lohstroh
Location: Frederik-Paulsen-Hörsaal

13:45 - 14:00: Dynamics of Confined Water in Carbon Nanohorns And Porous Magnesiuim Carbonate (ID 258) Antti Johannes Soininen, Marie-Sousai Appavou, Katsumi Kaneko, Tomonori Ohba, Sara Frykstrand, Ken Welch, Marina Khaneft, Maria Strømme, Marie-Claire Bellissent-Funel, Joachim Wuttke

14:00 - 14:15: Proton Disorder in D2O - Ice: A Neutron Diffraction Study (ID 149) K. Siemensmeyer, J.-U Hofmann, S.V. Isakov, B. Klemke, R. Moessner, D.J.P. Morris, D.A. Tennant

14:15 - 14:30: Mechanical Properties of Multiferroic Bi2Mn4O10: Full Set of Elastic Constants Determined by Inelastic Neutron Scattering (ID 161) Fabian Ziegler, Holger Gibhardt, Oleg Sobolev, M. Mangir Murshed, Thorsten M. Gesing, Götz Eckold

14:30 - 14:45: Phonon Renormalization in LaCoO3 by Inelastic Neutron Scattering (ID 150) Maximilian Kauth, John-Paul Castellan, John Mitchell, Klaus-Peter Bohnen, Frank Weber


15:00 - 15:15: Influence of Planar Defects in the Octahedral Tilt Structure on the Ferroelectric Properties of a Na1/2Bi1/2TiO3-based Perovskite (ID 158) Florian Pförr, Márton Major, Uwe Stuh, Bertrand Roessli, Wolfgang Donner
Dynamics of Confined Water in Carbon Nanohorns And Porous Magnesium Carbonate

Antti J. Soininen, Marie-Sousai Appavou, Katsumi Kaneko, Tomonori Ohba, Sara Frykstrand, Ken Welch, Marina Khanef, Maria Strømme, Marie-Claire Bellissent-Funel, Joachim Wuttke

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Keywords: confined water, dynamics

We have studied the dynamics of water confined in carbon nanohorns and in a magnesium carbonate material, upsalite, using the neutron backscattering spectrometer SPHERES at MLZ, Garching. Carbon nanohorns are analogous to carbon nanotubes except for different topology thus providing more heterogeneous confining environment. Upsalite, on the other hand, is a novel mesoporous material with exceptional water adsorption properties. In the case of carbon nanohorns, we studied the acquired spectra over the whole accessible $q$ range in contrast to what seems the contemporary standard procedure. A rigorous analysis of the $q$ and temperature dependencies of the relaxation times revealed a transition from local dynamics to more long range displacements near 225 K. Contradicting the fragile-to-strong crossover hypothesis, the transition was not thermodynamical, as shown by the $q$ dependency of the transition temperature. Above around 250 K, the quasielastic line broadened to a degree which prevented unambiguous fitting in the time window of the spectrometer. However, a weak Lorentzian with temperature independent width was present at these temperatures. This line was attributed to functional groups at the surface of the nanohorns. On the other hand, water in upsalite showed different behaviour to nanohorns and other common matrix material, such as MCM-41. The acquired spectra were not stretched but could be fitted with a single Lorentzian plus a delta line over a much broader temperature range, up to 360 K. At low temperatures, degeneracy between the delta line and the Lorentzian rendered observation of the usual super-Arrhenius behaviour of relaxation time impossible. Surprisingly, the dynamics did not escape the narrow time window of SPHERES at high temperatures. The slow dynamics even above the melting point of water were attributed to unusually high amount of bound water and partial crystallization of the matrix material during the experiment.

References:
Proton Disorder in D₂O – Ice: A Neutron Diffraction Study

K. Siemensmeyer¹, J.-U. Hofmann¹, S. V. Isakov², B. Klemke¹, R. Moessner³, J. P. Morris⁴, D. A. Tennant⁵

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Keywords: ice rules, proton disorder, spin ice

Water ice (H₂O) at low pressure can be understood as a hexagonal structure where each Oxygen atom is bound to two Hydrogen atoms. Further, each bond between Oxygen atoms is occupied by only one Hydrogen atom. These are the famous ice-rules that give rise to a highly disordered ground state with a residual entropy of \( R \ln(3/2) \). We have measured the diffuse scattering from a large D₂O – ice crystal using neutron diffraction. Different to previous descriptions of the ice-structure by Monte Carlo methods we are able to explain the data using an analytical method: The structure is mapped to a divergence-free dipolar model which is solved within a large-N approach. We obtain remarkable agreement between model and neutron results. The correlation length obtained at \( T = 30 \, K \) seems to be limited by the instrumental resolution.

References:
Mechanical Properties of Multiferroic Bi$_2$Mn$_4$O$_{10}$: Full Set of Elastic Constants Determined by Inelastic Neutron Scattering

Fabian Ziegler$^1$, Holger Gibhardt$^1$, Oleg Sobolev$^1$, M. Mangir Murshed$^2$, Thorsten M. Gesing$^2$, Götz Eckold$^1$

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Keywords: inelastic neutron scattering, elastic constants, multiferroics, velocity of sound

Motivation: Mullite-type Bi$_2$Mn$_4$O$_{10}$ [1] is an example for a multiferroic compound with a Néel temperature of 39 K, although its crystal structure [2] is not typical for allowing ferroelectricity. Both its nuclear and magnetic structural features differ from those of other rare-earth members of the R$_2$Mn$_4$O$_{10}$ family [3]. Mechanical distortions are frequently found to play important roles during ferroelectric phase transitions. Therefore, the knowledge of the elastic behavior seems crucial for a profound understanding of the interplay of magnetic and electric ordering behavior. Hence, we have undertaken an inelastic neutron scattering study on single crystals of Bi$_2$Mn$_4$O$_{10}$ aiming at the determination of the full set of nine elastic constants $c_{ij}$ provided by the orthorhombic system (Pbam).

Experimental setup: The inelastic neutron scattering experiments have been performed at the three-axes spectrometer PUMA@FRM II in Garching. The dispersion curves of the acoustic phonon branches in the Brillouin zone have been measured at room temperature for different propagation directions close to the zone center (Γ-point) and for different polarization vectors. The linear slopes of the dispersion curves are connected with a specific combination of the $c_{ij}$ depending on the propagation directions and the polarization vectors of the modes.

Results: We were able to determine all nine elastic constants $c_{ij}$ provided by the orthorhombic crystal. These results show a clear mechanical anisotropy indicating that the crystallographic a-axis is softer than the other principal directions. This behavior is associated with the mutual linking of the individual building blocks of the unit cell. Since the magnetization in the low-temperature phase is also oriented along the a-axis, this can be regarded as an indication for the important role of the magnetoelastic coupling for the existence of multiferroicity. Furthermore, some interesting resolution effects were observed allowing the detection of a phonon that does not obey the polarization selection rule.

References:
Phonon Renormalization in LaCoO₃ by Inelastic Neutron Scattering

Maximilian Kauth¹, John-Paul Castellan¹, John Mitchell³, Klaus-Peter Bohnen¹, Frank Weber¹

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Keywords: inelastic neutron scattering, phonons, dynamics

LaCoO₃ is investigated since the 1950’s because of its different spin states which are supposed to exhibit complex orders in certain temperature ranges. The material shows two broad magnetic-electric transitions, a diamagnetic to paramagnetic spin-state transition at T_{SS} ≈ 100 K and a metal-insulator transition at T_{MI} ≈ 500 K. The spin transitions on heating are proposed to be as follows: from a homogeneous low spin (LS) state to a mixed LS/HS state with strong spin-charge fluctuations at T = T_{SS} and, subsequently, into a homogeneous high spin (HS) state at T = T_{MI}. The lattice participates in the state mixture by expansion of CoO₆ octahedra around the HS sites, while the ones around LS sites have a reduced size. The originally proposed static order has not been observed experimentally and, hence, the ordering is expected to be dynamic and short-ranged (see figure 1).

We have investigated the lattice dynamical properties of LaCoO₃ using inelastic neutron scattering. Based on detailed ab-initio lattice dynamical calculations (performed in our institute), we aim for a comprehensive understanding of lattice dynamics in LaCoO₃. The above discussed crossovers and spin state order should be reflected in the lattice degrees of freedom via quasi elastic scattering and phonon renormalization effects.

Figure 1: The figure shows the expansion of the CoO₆ octahedra around the high spin sites and the size reduction of the Co³⁺ atoms in the low spin configuration (Co: blue spheres, O: green, La: grey). This breathing-type distortion (indicated by red arrows) is proposed to be dynamic and short-ranged. It is expected that the lattice distortion and the dynamics can be observed with inelastic neutron scattering around the wave vector q = (½, ½, ½), i.e. the unit cell is doubled along all three major cubic axes.

References:
Study of the Critical Fluctuations at the Curie Point in Iron Using the Longitudinal MIEZE Setup at RESEDA

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Keywords: neutron resonance spin echo, modulation of intensity with zero effort, fluctuations

We report the implementation of a new neutron spectroscopy technique with sub-µeV resolution based on the longitudinal neutron resonance spin-echo (LNRSE) technique at the beam-line RESEDA at MLZ in Garching, Germany. First proof of principle experiments show a dramatic increase in dynamic range with respect to classical transverse NRSE. In addition to standard LNRSE, the new setup allows to perform experiments using modulation of intensity with zero effort (MIEZE)\textsuperscript{1}. The LNRSE option may be used to access a wide range of momentum transfer and high energy resolution. In addition, the MIEZE setup allows investigating samples under depolarizing conditions, e.g. hydrogen-containing materials, ferromagnetic samples or studies in the presence of large magnetic fields, without loss of energy resolution.

As an example, we present a study on the archetypal ferromagnet iron using the MIEZE technique\textsuperscript{2}. We present a comprehensive study of the critical magnetic fluctuations around the transition from the ferro- to the paramagnetic phase. The MIEZE technique allows us to simultaneously extract the lifetime of the critical fluctuations over a wide $q$-range. Our comprehensive data is in qualitative agreement with the data from Mezei\textsuperscript{3} that was obtained by using the conventional spin-echo technique. The results demonstrate the importance of the dipolar interactions when describing this textbook example of a continuous phase transition.

References:
Influence of Planar Defects in the Octahedral Tilt Structure on the Ferroelectric Properties of a Na$_{1/2}$Bi$_{1/2}$TiO$_3$-based Perovskite

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Keywords: ferroelectric, diffuse scattering, nanostructure

Ferroelectric materials are used in many applications, e.g. as ultrasonic transducers. The most commonly used ferroelectrics like Pb$_x$Zr$_{1-x}$TiO$_3$ contain lead, which makes them potential hazards for human health and our environment. Consequently, lead-free ferroelectrics are currently being developed with the aim of replacing the lead-containing materials in the medium term. However, the microscopic mechanisms which determine the ferroelectric properties have to be identified before the properties can be optimized for specific applications.

Among the lead-free ferroelectrics, (1-x)Na$_{1/2}$Bi$_{1/2}$TiO$_3$-xBaTiO$_3$ is one of the most promising material systems. The dielectric properties around x = 0.06 were cited as $d_{33} = 125$ pC/N and $\varepsilon_{33}/\varepsilon_0 = 580$ [1]. These values are comparable to those of commonly used lead-containing ferroelectrics. However, the atomistic mechanisms leading to the relaxor properties are still unclear. Previous diffuse x-ray scattering experiments performed by our group on 0.96Na$_{1/2}$Bi$_{1/2}$TiO$_3$-0.04BaTiO$_3$ revealed features related to the local octahedral tilting order and planar defects separating different tilt domains [2]. These features react strongly to the application of an external electric field and their temperature dependence is clearly correlated with the dielectric permittivity.

We have complemented our x-ray scattering experiments with an elastic diffuse neutron scattering study [3]. Our results show the coexistence of multiple tilt systems over a wide temperature range and a strong temperature dependence of the respective domain sizes. On this basis, we propose a model of the nanostructure featuring chemically pinned tetragonal platelets in a rhombohedral matrix. The different tilt domains are separated by a cubic intermediate phase. Furthermore, a strong temperature dependence of the planar defect density was found, which peaks at the depolarization temperature.

References:
M1_2: Minisymposium
“Proteins: Dynamics and Interactions”, part 2

Tuesday, Sept. 20, 2016, 13:45 – 15:15. Session Chair: Tilo Seydel
Location: Hörsaal C

13:45 - 14:10: Influence of Pressure on Proteins Structure and Dynamics Investigated by Neutron Scattering (ID 273) Appavou Marie-Sousai

14:10 - 14:35: Exploring the Physical Behavior of a Novel Membrane Ultrastructure Involved in High Pressure Adaptation in Archaea (ID 272) Phil M. Oger

14:35 - 14:50: Effects of Crowding, Osmolytes, Temperature and Pressure on the Interaction Potential, Dynamics and Phase Behavior of Dense Protein Solutions (ID 269) Roland Winter

14:50 - 15:05: Internal Dynamics in the Denatured Bovine Serum Albumin Protein (ID 151) Felix Ameseder, Andreas Stadler, Aurel Radulescu, Olaf Holderer, Dieter Richter

15:05 - 15:15: Animated Symposium Discussion Session. Tilo Seydel
Influence of pressure on proteins structure and dynamics investigated by neutron scattering.

Appavou Marie-Sousai.

Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science JCNS Outstation at MLZ, Lichtenbergstraße 1, 85747 Garching, GERMANY

Pressure is a physical parameter which influence is widely spread in Earth. Indeed, micro-organisms like *Bacillus Licheniformis* can live in Deep Ocean until 10 km and they can sustain pressure of about 1000 bar [1]. Multimeric protein subunits association-dissociation phenomena and protein folding and unfolding is also more studied using this unusual parameter. The interest of using high pressure comes for its use in food industries for sterilization process since this procedure has less influence on texture and taste than thermal sterilization. In medicine, high pressure is studied for bio-conservation of blood or other oxygen transporter molecules for example

Small angle neutron scattering is a suitable tool to investigate soft matter material: for example, proteins from their native to their fully unfolded state since the technique is not destructive and model based on polymer theory studies can be adapted to characterize the structures. The JCNS at MLZ has a 5000 bar high pressure with sapphire windows especially dedicated for structural investigation of soft matter, and particularly biology, investigated by Small Angle Neutron Scattering. The design is based on the high pressure set up described in Kohlbrecher et al 2007 [2]. The cell will be described and a typical example will be presented.

Quasielastic neutron scattering allows to explore at the atomic space scale internal motions of proteins in the pico-second time scale [3,4]. Few neutron scattering studies on biomolecules were performed up to date. The first unfolding study using inelastic neutron scattering was published by Doster et al [5,6]. Other studies have been performed by Appavou et al for BPTI [7] and by Di Bari for Trypsin [8]. We would like to present a series of results as a function of pressure we have obtained with the TOFTOF time of flight quasielastic neutron scattering spectrometer (Garching, Germany). We have built a high pressure cell which can sustain a pressure of 2000 bar. The high transmission, due to the aluminium alloy of the cell, allows us to make a quite accurate observation of the influence of pressure on the translational diffusion and the internal dynamics of haemoglobin.

References

Exploring the physical behavior of a novel membrane ultrastructure involved in high pressure adaptation in archaea

Phil M. Oger

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Most of Earth’s biotopes are located in the deep-sea and the deep subsurface under elevated hydrostatic pressures (HHP) inhibitory to surface organisms. Experimental evidence on macromolecules shows that HHP has a different impact depending on the biological macromolecule, increasing or decreasing stability, although HHP has a similar negative impact on the cellular function. In many ways the effects of HHP resemble that of an increase or a decrease in temperature, such as protein denaturation, membrane destabilization, alteration of transcription and translation. Biological membranes play an important role as a barrier from the environment and host numerous cellular processes. They are highly sensitive to temperature (T) and pressure (P), which strongly impact membrane fluidity by modifying membrane permeability and membrane-associated functions. To adapt to T or P fluctuations, bacteria maintain optimal membrane fluidity by altering the degree of unsaturation and the chain length of fatty acids, a mechanism named homeoviscous adaptation. Structural differences between archaeal membrane lipids (containing glycerol ether bonds and phytanyl chains) and bacterial analogues (containing fatty acids esterified to glycerol) raise the question of membrane fluidity regulation and membrane behavior in hyperthermophilic and piezophilic archaea.

We have characterized the lipid membrane composition of the piezophilic archaeon Thermococcus barophilus by HPLC/APCI-MS. Archaeol (diether isoprenoid lipid) and caldarchaeol (tetraether isoprenoid lipid) are the major core lipids of T. barophilus. The apolar lipids of the strain consist in series of C35 and C40 unsaturated isoprenoid hydrocarbons with a lycopane-like carbon skeleton. Variations in P or T relative to optimal conditions of growth induce a modification of the relative proportions of archaeol and caldarchaeol. T. barophilus increases its proportion of caldarchaeol at high T and low P and decreases it at low T and high P. In parallel, the degree of unsaturation of the lycopane-chains increased with a decrease of T or an increase of P. This lipid composition indicates that T. barophilus possesses a mixed monolayer-, bilayer-type membrane. These results strongly suggest that apolar lipids are membrane structural lipids (Cario et al. 2015). We propose a model according to which apolar lipids are incorporated at the interface between the two layers, where they play a central role in membrane rigidity and in the regulation of the proton gradient across the membrane.

To characterize the behavior of such a novel membrane ultrastructure, we have used a synthetic membrane analog composed of two polar archaeal lipids, Diphytanoylphosphatidyl choline (DPhPC) and Diphytanoylphosphatidyl ethanolamine (DPhPE), and a fully saturated apolar isoprenoid lipid, squalane (C62H62) or fully deuterated squalane (C62D62). The localization of the squalane molecule was determined by H/D contrasting in neutron diffraction data acquired on multilayer oriented membrane prepared on silicium wafers. Data was acquired at the D16 spectrometer at ILL in Grenoble. Our preliminary data demonstrated without any doubt the ability of apolar lipids to insert in the midplane of an archaeal lipid bilayer. Preliminary results using IR spectroscopy on the same lipid mixture confirm that the insertion of apolar lipids further increases P and T stability of the membrane as evidence from the shifts of the phase transition. Furthermore, the presence of squalane increases the amplitude of the phase transition, indicating a stronger rearrangement of lipid molecules at the transition. We are still in the process of characterizing the full behavior using solid state NMR, IR spectroscopy, DSC, Neutron Diffraction and molecular dynamics, using both synthetic and natural lipid mixes. Recent developments and their implications for the function of the membrane will be discussed.


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Keywords: High Hydrostatic Pressure, Adaptation, Piezophiles, Hydrothermal Vents, membrane.
Effects of Crowding, Osmolytes, Temperature and Pressure on the Interaction Potential, Dynamics and Phase Behavior of Dense Protein Solutions

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Keywords: high pressure, proteins, interaction potential, osmolytes, phase diagram

Mechanisms have evolved in Nature that allow living organisms to deal with extreme environmental conditions, for example by producing particular organic osmolytes. For example, certain marine organisms living in the deep sea have evolved a surprising simple mechanism to counteract the deleterious effects of the cellular waste product urea and of hydrostatic pressure by trimethylammonium N-oxide. We investigated the effect of pressure on the structure and intermolecular interactions of dense lysozyme solutions in various cosolvent mixtures and upon addition of various Hofmeister anions using small-angle X-ray scattering in combination with liquid-state theoretical approaches. Supplementary thermodynamic information was obtained by employing calorimetric techniques, densitometry and ultrasound velocimetry. We show that the particular structural properties of water, specific ion effects, and solvent-mediated processes play a crucial major role in protein stabilisation, notably under high hydrostatic pressure conditions. Also the effect of confinement on the solvational properties, intermolecular interaction and dynamic properties of proteins was studied, including the effects of self-crowding and macromolecular crowders on the temperature-pressure stability diagram of proteins. We also discuss the effect of pressure on the second virial coefficient $B_{22}$ and how pressure can be used to control and fine-tune protein crystallization. Moreover, we present results on the phase behavior of dense lysozyme solutions in the liquid-liquid phase separation region, and characterize the underlying intermolecular protein-protein interactions as a function of temperature and pressure in this region of phase space. A re-entrant liquid-liquid phase separation region has been discovered at elevated pressures, which originates in the pressure dependence of the solvent-mediated protein-protein interactions.

References:
Internal Dynamics in the Denatured Bovine Serum Albumin Protein

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Keywords: protein, structure, dynamics

The process of protein folding is highly dependent on the amino acid composition as well as on the solution condition, especially on the presence of denaturant. Our approach is to describe the dynamics and structure of the unfolded protein using polymer theory, and measure the folding as a function of denaturant type and denaturant concentration which has proved to be promising in single-molecule Förster resonance energy transfer FRET experiments\textsuperscript{1,2}.

Here, we use small-angle neutron scattering to determine the structure of bovine serum albumin BSA in solution and at various concentrations of guanidine hydrochloride and beta-mercaptoethanol. The center of mass diffusion of native and denatured BSA is investigated with dynamic light scattering spectroscopy. Neutron spin-echo spectroscopy is used to cover a time range up to 140ns.

Small-angle scattering results of native BSA are in agreement with the crystal structure model of a respective monomer\textsuperscript{3} whereas the denatured protein was found to be internally disordered similar to a swollen polymer chain.

Neutron spin-echo spectroscopy of denatured BSA reveals a significant contribution of internal dynamics to the overall global diffusion that is clearly missing in the native state.

We successfully showed that models from polymer theory are suitable for the interpretation of the observed motions in agreement with results obtained from mentioned FRET experiments\textsuperscript{1,2}. While BSA at 6M guanidine hydrochloride follows quasi pure Zimm dynamics, dynamics of BSA at 4M guanidine hydrochloride are interpreted with a Zimm model including internal friction (ZIF) that reveals an offset of 18.5ns is required as contribution to all relaxation times.

References:

M2_2: Minisymposium “High brilliance compact neutron sources – status and perspectives”, part 2

Tuesday, Sept. 20, 2016, 13:45 – 15:15. Session Chair: Thomas Gutberlet
Location: Hörsaal D

13:45 - 14:05: Status of Neutron Imaging with Respect to Material Research with Neutron Sources of Limited Intensities (ID 285) Eberhard H. Lehmann

14:05 - 14:25: Probing Multi-functional Oxides by Neutron Scattering (ID 291) Manuel Angst

14:25 - 14:45: Neutron Scattering Studies of Proteins: Status and Perspectives (ID 284) Frank Schreiber

14:45 - 15:05: Neutron Scattering for Studying Stimuli Sensitive Polymer Coatings (ID 270) Regine von Klitzing
Status of Neutron Imaging with respect to Material Research with neutron sources of limited intensities

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The methodical progress in the neutron imaging technologies has been demonstrated successfully at several prominent neutron sources. New installations have started and upgrade measures are planned. Although high beam intensity is very welcome to be able to perform with high spatial resolution, time resolution and high image quality, the high efficiency of modern digital imaging detectors enable short acquisition time even at moderate intensity (e.g. TRIGA AI Vienna at 250 kW).

While the “flagship installations” at HZB, TUM or PSI are heavily overbooked by scientific and industrial customers, sources like ILL, ISIS and FLNP Dubna just started some activities on different level.

Next to neutron radiography and tomography where the majority of investigations are performed routinely, new attempts with grating interferometry, energy-selective imaging, polarized neutron imaging have been introduced and started to be implemented into the user program. An important way to compete with X-ray imaging methods is the improvement of the spatial resolution down to a few micro-meters and the flexibility for contrast variations by selecting the most suitable neutron energy. The study of relevant materials (H, Li, B, …) in bulky metallic environment enable unique studies for new energy concepts and materials like batteries. Since the most metals are transparent for neutrons their inner properties can be observed even under low neutron intensities in reasonable time.
Probing Multi-functional Oxides by Neutron Scattering

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Keywords: neutron, functional oxides, correlated electrons, multiferroics

Complex transition-metal oxides are often correlated electron systems with ordering tendencies of electronic degrees of freedom (charge, orbital, spin) potentially leading to functionalities interesting for applications. Functional oxides include for example magnetoelectric multiferroics, which have a large applications potential in information technology. Suitable mechanisms of multiferroicity encompass ferroelectricity driven by charge order (CO) and spin order (SO), both of which are highly suited for investigations by scattering methods. SO-driven ferroelectricity involves naturally a very strong magnetoelectric coupling. Ferroelectricity arising from CO is also expected to yield large magnetoelectric coupling as the same ions are involved in both CO and SO.

Neutron scattering is particularly well-suited to elucidate the often complex SO indispensable to understand magnetoelectric coupling and multiferroic mechanisms. However, x-ray scattering is often advantageous for CO, not least because smaller and tendencielly more perfect crystals [1] can be used. Furthermore, the relevant CO and SO are often long-period structures that necessitate a high resolution in reciprocal space. Based on example materials investigated by neutrons and/or x-rays, I will discuss the benefits of a brilliance-optimized source.

I will present two case studies of oxides proposed to exhibit CO-based multiferroicity and one example of SO-based multiferroicity:

1) Rare earth ferrites had long been considered the prototypical “proof-of-principle” examples and have correspondingly attracted a lot of attention [2]. However, the CO, determined with scattering techniques [3], turned out to be non-polar, and previous macroscopic indications of ferroelectricity have been explained in terms of extrinsic effects.

2) Classical magnetite both shows macroscopic indications of at least relaxor ferroelectricity and possesses a polar CO crystal structure [4]. Recent time-resolved diffraction data con-firm a structural response to voltage-pulses consistent with ferroelectric switching.

3) Spin-spiral-based multiferroics have been extensively investigated. Due to the high temperature scale most attractive are compounds of the hexaferrite family, but in many cases the spin structure of the ferroelectric phase is not solved. I will discuss mapping of chiral domains in a classic hexaferrite, which indicates a spin structure consistent with spiral-based ferroelectricity.

References:
Neutron Scattering Studies of Proteins: Status and Perspectives

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Keywords: neutron, instrumentation, structure, dynamics, proteins, solutions, liquids, crystallization

Proteins are considered the machines of life. Their structure, dynamics, and interactions are crucial for many biological processes on the molecular scale. Importantly, these take place in a complex environment, for which the solvent (water), various ions, and other proteins are important ingredients. Studying and quantifying protein behavior under these conditions, frequently characterized by macromolecular crowding, i.e. high concentrations, is a challenging task, to which neutrons can make important contributions.

Here we discuss status and perspectives of neutron scattering from proteins. We cover both elastic scattering with a focus on small-angle scattering to address structural issues as well as interactions and inelastic / quasi-elastic scattering to address the dynamics over a broad range of time scales using TOF, NBS, and NSE techniques.

After a general overview we present some selected recent examples related to charge-modified interactions of proteins. This route for tailoring the interaction potential is exploited for controlling a) crystallization, b) gelation and amorphous aggregation, as well as c) smaller aggregate formation including their dynamics and kinetics.

While protein crystallization is a precondition for most of the progress in structural biology and pharmacy, it is still largely impossible to predict it reliably. We present a study of protein crystallization induced by multivalent ions monitored by simultaneous small-angle and Bragg scattering in real time. From the real-time observation of the crystallization kinetics, we deduce a multistep crystallization mechanism, with an intermediate phase as a “precursor”, evidenced by small-angle “cluster” features, followed by the nucleation of crystals. We also discuss issues related to arrested states, which appear to be an aggregation pathway competing with crystallization. We outline concepts for controlling and understanding protein aggregation pathways and the branching between them in aqueous solution by addition of multivalent ions.

Second, we discuss complementary investigations of the dynamics of these systems using quasi-elastic neutron scattering, showing a remarkably universal behavior of the effective diffusion as a function of concentration and salt under suitable conditions.

Finally, we comment on the use of complementary techniques including X-ray and light scattering as well as the use of present and future neutron sources.

Invaluable contributions by A. Sauter, F. Zhang, F. Roosen-Runge, T. Seydel, M. Grimaldo, and many others as well as fruitful collaborations at the FRM2 and the ILL are gratefully acknowledged.

References:
Neutron Scattering for Studying Stimuli Sensitive Polymer Coatings

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Keywords: Polymer coating, stimuli sensitive coatings, reflectometry, GISANS, GINSE

For fabrication of stimuli responsive coatings one challenge is to generate stable films which are still mobile and sensitive to outer parameters. The talk will focus on 2 different types of thin polymer films at solid interfaces: 1) films formed by deposition of hydrogel microgels and 2) polymer brushes. Both architectures have in common that they consist of N-isopropylacrylamide (NIPAM) monomers. During the last decades microgels made of N-isopropylacrylamide (NIPAM) have attracted much interest and were studied by several techniques like microscopy and light scattering. These polymer particles show thermoresponsive behaviour and can therefore be classified as “smart” materials. By copolymerisation with organic acids such as acrylic acid (AAc) the temperature of the volume phase transition as well as the swelling ratio can be influenced. Moreover charged copolymers are sensitive to changes in pH and ionic strength. Our work focuses on the fabrication of stimuli responsive films and on the effect of geometrical confinement on the phase volume transition of these microgel particles [1]. The effect of cross-linker and co-monomers on the swelling behaviour and on the elasticity is presented [2]. The second example is coating with PNIPAM brushes synthesized via ATRP and grafting from method [3]. Beside pH, temperature light is a very efficient stimulus, since it can trigger quite fast and local the volume phase transition. Therefore additives like surfactants with azobenzene groups [4] and gold nanoparticles [5] are embedded within both microgels and brushes. In case of gold nanoparticles, the change in optical properties of microgels and brushes and the impact of gold nanoparticles as hot spots is studied. For a better understanding of the structure-function relation it is essential to get insight into the distribution of the gold nanoparticles within the polymer coatings. Therefore neutron and x-ray reflectometry are suitable methods [3,7]. Adsorption at the interface affects the volume phase transition due to the compression of the gel structure. In this context we studied the structure and the dynamics of the gels in bulk and at the interface with SANS and NSE in bulk and under grazing incidence (GISANS, GINSE) [6,7].

Session 3: Instrumentation I / Magnetism I

Tuesday, Sept. 20, 2016, 16:00 - 17:30. Session Chair: Oliver Stockert
Location: Frederik-Paulsen-Hörsaal

16:00 - 16:15: **Beamline for European Materials Engineering Research (BEER) at the ESS (ID 110)** Jochen Fenske, Mustapha Rouijaa, Gregor Nowak, Reinhard Kampmann, Peter Staron, Heinz-Günter Brokmeier, Martin Müller, Andreas Schreyer, Markus Strobl, Jan Saroun, Premysl Beran, Jan Pilch, Petr Sittner, Pavel Strunz, Victor Ryuthkin, Lukas Kaderavek, Petr Lukas

16:15 - 16:30: **C-SPEC- A Cold Time of Flight Spectrometer for the ESS (ID 184)** Wiebke Lohstroh, Pacale Deen, Joseph LeBouffy, Stephane Longeville, Jean-Marc Zanotti, Christiane Alba-Simionesco, Jürgen Neuhaus, Winfried Petry


16:45 - 17:00: **Neutron Diffraction on the Frustrated Spin-Chain Linarite at Low Temperatures and High Fields (ID 167)** Leonie Heinze, Britta Willenberg, Jens-Uwe Hoffmann, Anja U.B. Wolter-Giraud, Bernd Büchner, Kirrily C. Rule, Andrew Studer, Bachir Ouladdiaf, Stefan Süllow

17:00 - 17:15: **Static and Dynamic Properties of the Spiral Magnet Ba2CuGe2O7 Studied by Neutron Resonance Spin Echo Spectroscopy, Small Angle Neutron Scattering and Triple Axis Spectroscopy (ID 176)** Sebastian Mühlbauer, Jonas Kindervater, Wolfgang Häußler, Georg Brandl, Martin Mansson, Markus Garst

Beamline for European Materials Engineering Research (BEER) at the ESS

Jochen Fenske,1 Mustapha Rouijaa,1 Gregor Nowak,1 Reinhard Kampmann,1 Peter Staron,1 Heinz-Günter Brokmeier,1,2 Martin Müller,1 Andreas Schreyer,3 Markus Strobl,4 Jan Saroun,4 Premysl Beran,4,5 Petr Sittner,4 Pavel Strunz,4 Victor Ryuthkin,4 Lukas Kaderavek,4,5 Petr Lukas
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Keywords: neutron, instrumentation, engineering

The continuous development of advanced structural materials and novel production processes are key for industrial manufacturing, e.g. in the transport and clean energy production sectors. Future successful research efforts will require understanding micro/nanostructure, residual stress evolution and phase transformations during processing and their role on the mechanisms that determine material and component performance. The upcoming Beamline for European Materials Engineering Research (BEER) to be built as part of the European Spallation Source (ESS) offers new opportunities for such investigations. The diffractometer combines the high brilliance of the long pulsed neutron source with high instrument flexibility. It includes a novel chopper technique that extracts several short pulses out of the long ESS pulse, leading to substantial intensity gain of up to an order of magnitude compared to pulse shaping methods for materials with high crystal symmetry. This intensity gain is achieved without compromising resolution. More complex crystal symmetries or multi-phase materials will be investigated by additional pulse shaping methods. The different chopper set-ups and advanced extracting techniques offer extremely broad intensity/resolution ranges, which today do not exist at any other engineering instrument. Furthermore BEER offers an option of simultaneous SANS or imaging measurements without compromising diffraction investigations. This flexibility opens up new possibilities for in situ experiments studying materials processing and performance under operation conditions. To full fill this task, advanced sample environments, dedicated to thermo-mechanical processing, are foreseen, e.g. a Gleeble® or a quenching and deformation dilatometer.

BEER has entered in 2016 the first phase of the construction phase. In this phase the instrument budget will be assigned and the design and scientific scope of the instrument will be revised in accordance with the budget. In this contribution we give an update on the design and performance of the engineering diffractometer BEER with regard to the assigned instrument budget.
C-SPEC - a cold time of flight spectrometer for the ESS

Wiebke Lohstroh, Pascale Deen, Joseph Guyon LeBouffy, Stéphane Longeville, Jean-Marc Zanotti, Christiane Alba-Simionesco, Jürgen Neuhaus, Winfried Petry

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Keywords: instrumentation

The European Spallation Source (ESS), expected to be the world’s most powerful neutron source, will begin operations in 2019. Among the endorsed instruments foreseen for day one instrumentation at ESS, is the cold time-of-flight spectrometer C-SPEC. C-SPEC is a joint proposal from the Technische Universität München, Germany, and the Laboratoire Léon Brillouin, Saclay, France. The high performance cold time-of-flight-spectrometer will serve multiple disciplines to address scientific questions from hard and soft matter research, life sciences, geosciences or magnetism. C-SPEC will address time dependent phenomena under realistic conditions. While current day spectroscopic experiments mainly probe the static state of a sample, neutron spectroscopy experiments that probe the time dependent behaviour, e.g. of electrolytes in an electric field, or laser excited light harvesting proteins, are still in an exploration stage, mainly due to the lack of flux at the instruments available today.

C-SPEC benefits from the high brilliance of the ESS spallation source in addition to the use of a novel chopper implementation and as such will address scientific questions raised by probing time dependent phenomena. The unique pulse structure of the ESS with its long pulse duration (2.86 ms) and a repetition rate of 14 Hz requires new concepts for the instrumentation to make optimum use of the available source time frame. With an instrument length of ~160 m, a wavelength range of $\lambda \leq 2$ Å can be probed within each ESS time period via rate–repetition mode. The energy resolution can be tuned in the range of $\Delta E/E = 6 - 1\%$, and C-SPEC will utilize cold neutrons in the range from $\lambda = 2 - 15$ Å with the focus on the cold part of the spectrum. The guide is optimised to enhance signal to noise and will be able to focus on samples ranging from several mm$^2$ to several cm$^2$ in area. The large detector area, with a radius of 4 m, 40 – 140 degrees and 4 m in height, typical on a chopper spectrometer will be designed with optimal energy and Q resolution in mind while maintaining the highest signal to noise ratio. C-SPEC is in the preliminary engineering phase and we will present the current design layout and the expected performance.
Application of Boron Carbide Coatings in Neutron Detectors

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Keywords: neutron detectors, Boron carbide coatings, MWPC, instrumentation

Fundamental changes in neutron detection technology were triggered by the extremely limited availability and raising price for \textsuperscript{3}He. Modern neutron Multi-Wire-Proportional-Chambers (MWPC) based on \textsuperscript{10}B\textsubscript{4}C coatings in inclined geometry are distinguished by their position resolution, which surpass by one order of magnitude that one of \textsuperscript{3}He tubes at comparable detection efficiency [1]. Thin-film preparation and analysis of \textsuperscript{10}B\textsubscript{4}C coatings came in to the fore of the R&D activity for these novel detectors. This activity was performed as an in-kind contribution to the ESS instrumentation, and was part of the German support to the ESS Pre-Construction Phase and Design Update [2]. Neutron conversion layers of \textsuperscript{10}B\textsubscript{4}C have been developed and deposited with thicknesses of up to 10 μm on pretreated Al substrates (300 μm thickness) with thickness uniformity better than 2 % over the entire deposition area of 1430 mm x 100 mm [3]. The \textsuperscript{10}B\textsubscript{4}C coatings show excellent adhesion to the flexible substrate even under strong externally applied stress or strain. The chemical and isotopic compositions of the \textsuperscript{10}B\textsubscript{4}C coatings were investigated by means of XPS, SIMS, and RBS and are in the range of requirements or better [1]. The manufactured coatings have been applied in two neutron detector concepts based on inclined and perpendicular neutron incidence on the converter coating. Measurements on neutronic properties, which were obtained by ToF-experiments at REFSANS in MLZ (Munich) show a quantum efficiency of up to 90 % (compared to an 1 inch \textsuperscript{3}He tube (10 bar)). This efficiency was measured for a 1.2 μm thick \textsuperscript{10}B\textsubscript{4}C converter coating at small angles of incidence (\textit{α}_{i} = 1 deg.) of the neutron beam with respect to the converter surface. The position resolution achieved by the investigated detector concepts ranges from 4 mm down to the sub-mm range. The investigated detector concepts have the potential to replace \textsuperscript{3}He detectors and are planned to be utilized at the upcoming diffraction beamlines at the ESS e.g. engineering diffractometer BEER.

References:


Neutron Diffraction on the Frustrated Spin-Chain Linarite at Low Temperatures and High Fields

Leonie Heinze, Britta Willenberg, Jens-Uwe Hoffmann, Anja U. B. Wolter-Giraud, Bernd Büchner, Kirrily C. Rule, Andrew Studer, Bachir Ouladdiaf, and Stefan Süllow

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Keywords: spin-chain, quantum magnet, magnetic frustration

The natural mineral linarite, PbCuSO₄(OH)₂, has been established as a model compound of the frustrated one-dimensional spin-chain with ferromagnetic nearest-neighbor and antiferromagnetic next-nearest-neighbor interaction [1]. Within the last years, it has been demonstrated that it exhibits a complex magnetic phase diagram in applied fields along the crystallographic b axis for temperatures below 2.8 K (see Fig. 1). The ground state phase I exhibits helical order, phase IV has been shown to be antiferromagnetic, in phase III helical and antiferromagnetic coexist. Recent neutron diffraction and NMR measurements with focus on phase V have revealed a complex spin density wave phase with an incommensurability vector [0 k y 0.5] shifting with field [2].

Here, we present new neutron diffraction measurements with special emphasis on the low temperature/high field regime of the magnetic phase diagram of linarite. From these measurements, the temperature and field dependence of the magnetic moment per Cu atom was established for temperatures down to 50 mK and magnetic fields \( B \parallel b \) up to 9.4 T. As well, the nature of the phase transition from phase V into phase IV being of first order was established. In addition, we find that a shift of the incommensurability vector in phase V occurs not only with magnetic field but also with temperature.

References:
Static and Dynamic Properties of the Spiral Magnet Ba$_2$CuGe$_2$O$_7$ Studied by Neutron Resonance Spin Echo Spectroscopy, Small Angle Neutron Scattering and Triple Axis Spectroscopy

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Keywords: heli-magnetism, magnetic phase transitions, neutron spectroscopy, SANS

Abstract: Spiral magnetic structures have raised great scientific interest in the last decade, caused not only by their multiferroic and topological properties. As pointed out by Brazovskii, also the phase transition into spiral magnetic phases can be of special interest [1]: If critical magnetic fluctuations soften at finite momentum transfer an otherwise 2nd order phase transition may be driven first order. This scenario, recently found to be realized in the helimagnet MnSi [2], is relevant for incommensurate magnetic structures in general. As the Brazovskii scenario is exclusively based on phase space considerations it is an interesting open question whether it is also applicable to systems with lower dimension. A possible candidate for such considerations is the non-centrosymmetric tetragonal antiferromagnet (AF) Ba$_2$CuGe$_2$O$_7$ ($P4̅2_1m$), which shows an incommensurate spiral magnetic structure, provided by Dzyaloshinskii-Moryia interaction. Below $T_N=3.2$K and at zero magnetic field, neutron diffraction has established an incommensurate, almost AF cycloidal magnetic structure in the (a,b)-plane with $\xi=0.0273$. Weak inter-plane coupling leads to an essentially 2-D behavior close to $T_N$. Four magnetic satellite reflections indicative of two degenerate magnetic domains are observed at $(1\pm\xi, \pm\xi,0)$, centered at the AF Neel point (1,0,0) [3, 4]. The phase transition from paramagnetism to spiral long-range order is characterized by interplay of 2-D physics and Brazovskii correlations.

We use a combination of SANS, longitudinal NRSE and TAS for a detailed study of the magneto-structural properties of Ba$_2$CuGe$_2$O$_7$ and the phase transition from the helimagnetic to the paramagnetic phase. We find that the Brazovskii scenario is still realized in the effectively 2-D system [5]. In addition, we show how Larmor labeling on a NRSE spectrometer can be used for a unified approach to study both static and dynamic properties of incommensurate magnetic structures with small propagation vectors [6].

References:
Measurements of the magnon mass in Dzyaloshinskii-Moriya helimagnets by small-angle neutron scattering

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Keywords: chiral magnetism, small-angle (polarized) neutron scattering, spin wave dispersion

We developed the technique to study the spin wave dynamics of the Dzyaloshinskii-Moriya (DM) helimagnets by polarized small-angle neutron scattering. The cubic noncentrosymmetric structure of the B20 compounds produces a helical (homochiral) structure with the wave vector \( k_s = D/J \) balanced by the competition of the large ferromagnetic exchange interaction \( J \) and the small asymmetric Dzyaloshinskii-Moriya interaction \( D \). The energy difference between these two phases is experimentally accessible by the critical magnetic field \( H_{C2} \), which is needed to transform the helix into the field-induced ferromagnet state. The dispersion relation of the spin waves in the magnetic field above \( H_{C2} \) was given by Kataoka¹:

\[
\varepsilon = \mathcal{A} (q - k_s)^2 + \Delta H,
\]

where \( \Delta H = H - H_{C2} \) and \( k_s \) matches with the orientation of the external magnetic field. The sign of the DM constant determines the direction of the helix wave vector \( k_s \) being parallel or antiparallel with respect to the direction of the field. Compared to the dispersion relation of a ferromagnet, there are three significant differences: Firstly, the only minimum of the curve is shifted from the position \( q = 0 \). Secondly, the sign of the DM constant determines the preferred direction of the propagation of the spin waves. Thirdly, the spin wave gap related to the magnetic field is shifted by the value of \( H_{C2} \). In this case small-angle scattering is limited within the cut-off angle \( \theta_C(H) = \theta_0 \mu \Delta H/E_N \). Due to the anisotropic dispersion relation the magnetic field must be set perpendicularly to the incident neutron beam in order to extract the maximal scattering contribution. Using small-angle neutron scattering we are able to detect the elastic peak at \( Q = k_s \). Applying a magnetic field \( H \) above \( H_{C2} \) the elastic peak vanishes and a circle of neutron scattering with the center at \( Q = k_s \) and a radius equal to \( \theta_C \) remains, which is only related to the spin wave stiffness \( \mathcal{A} \).

We will present two examples of studying the spin wave dynamic by small-angle neutron scattering in DM helimagnets: MnSi² as well as the high temperature phase of FeGe.

References:
M1_3: Minisymposium
“Proteins: Dynamics and Interactions”, part 3

Tuesday, Sept. 20, 2016, 16:00 – 17:30. Session Chair: Judith Peters
Location: Hörsaal C

16:00 - 16:25: On Hierarchical Dynamics of Proteins in Solution (ID 277)  
Felix Roosen-Runge

16:25 - 16:50: Dynamics of Partially Folded and Unfolded Proteins Investigated with Quasielastic Neutron Scattering (ID 278)  
Andreas Stadler

16:50 - 17:15: Probing Protein Energy Landscapes by Quasielastic Neutron Scattering (ID 274) Gerald Kneller

17:15 - 17:30: Animated Symposium Discussion Session. Judith Peters
On Hierarchical Dynamics of Proteins in Solution

Felix Roosen-Runge1, Marco Grimaldo1,2, Christian Beck2, Frank Schreiber2, Tilo Seydel1

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Keywords: dynamics, proteins, modelling, diffusion

The dynamics of proteins in solution depends on the interplay of environmental factors such as concentrations of protein and additives as well as temperature and pressure. Besides translational and rotational diffusion of the entire protein molecule, inter-domain motions, backbone fluctuations, and relaxations on the level of single amino acids and chemical groups contribute to a hierarchical diffusive process, mirroring the hierarchical structure of proteins. Here, we discuss recent results from neutron scattering and computer simulations on the hierarchical dynamics of proteins, aiming for a comprehensive understanding of dynamical consequences of macromolecular crowding and temperature, i.e. thermal activation of motions and thermal denaturation. Macromolecular crowding does not only slow down the global diffusion, but also affects the internal dynamics [1,2,3]. With changing temperature, protein motions are smoothly activated, until thermal denaturation presents a clear and sharp transition in global and internal dynamics [3,4]. Interestingly, the data can be modelled well with an analytical fit function representing the stochastic switching between two diffusive states [5]. Finally, the general implications for studies of hierarchical dynamics in protein solutions using incoherent quasi-elastic neutron scattering are discussed.

References:
Dynamics of Partially Folded and Unfolded Proteins Investigated with Quasielastic Neutron Scattering

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Keywords: quasielastic neutron scattering, protein folding, dynamics

Protein folding is a fundamental process in molecular biology. The protein folding transition occurs from the fully unfolded peptide chain via metastable and partially folded intermediates towards the stable and native folded structure at the end of the folding collapse. Myoglobin (Mb) serves as a model system in the field of biophysics as it is comparatively small, which allows to obtain an understanding of the underlying physical principles governing its dynamical behaviour, and it is still sufficiently complex to show typical protein behaviour. Apomyoglobin (apo-Mb) – myoglobin without the heme group – is less stable than Mb and can be trapped in different folded, partially folded molten globules and unfolded states under equilibrium conditions depending on the chosen solvent conditions.

I will present an investigation on the dynamics of the protein in its different folded states by quasielastic neutron scattering1,2. The samples have been measured in the solution state to allow for solvent induced effects and to enable reversible thermodynamic properties. Global protein diffusion and internal macromolecular dynamics could be separated from the recorded spectra. Detailed insight into the properties of the internal dynamics of the different folded states of the protein was obtained. The conformational entropy difference $\Delta S_{\text{conf}}$ between different folded conformations and the unfolded state could be determined and was compared to the entropy difference $\Delta S$ obtained from thermodynamic parameters. The observed difference between $\Delta S$ and $\Delta S_{\text{conf}}$ was attributed to the entropy difference $\Delta S_{\text{hydr}}$ of dynamically disordered water molecules of the hydration shell. Our results point out the relevance of conformational entropy of the protein and the hydration shell for stability and folding of myoglobin.

References:
[2] Stadler, Demmel, Ollivier, Seydel; Picosecond to Nanosecond Dynamics Provide a Source of Conformational Entropy for Protein Folding; submitted
Probing protein energy landscapes by quasielastic neutron scattering

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Keywords: quasielastic neutron scattering, proteins, energy landscapes, multiscale relaxation

Proteins are fundamental building blocks of biological systems and their functional dynamics is characterized by a large spectrum of characteristic time scales, ranging from sub-picoseconds to hours. Quasielastic neutron scattering can be used to probe stochastic motions of proteins on time scales between roughly 0.1 picoseconds and 100 nanoseconds. The dynamics seen in this time window is also accessible to molecular dynamics simulations, which enables in-depth analyses of the structural dynamics of proteins by corresponding “virtual experiments”. In this talk it will be shown how this approach can be used to model scattering functions in terms of relaxation rate spectra and how the latter can be translated into distributions of energy barriers describing the “energy landscape” of proteins [1]. Different models based on the concept of fractional Brownian Dynamics will be discussed in this context, relating them to the framework of the Generalized Langevin equation [2-5]. The second part of the talk is devoted to a new representation of neutron scattering functions in terms of neutron-induced transition probabilities between different levels of the protein energy spectrum. The formalism is the momentum-space analogue of the Frank-Condon theory for vibronic transitions in molecules due to the absorption or emission of photons.

References:
Poster Session 1

Posters will be up during the entire conference. During the first poster session, beer and snacks will be served. The second poster session is on Wednesday, 16:30-18:00. Software will not only be presented on posters, but also live on computers.

For the list of posters and abstracts, please refer to page 109 and following pages in the end of this document.
Das Neutron und das Universum

Stephan Paul
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Keywords: neutron, universe, particle physics


Der Vortrag gibt eine Einführung über die Beziehungen zwischen Laborbeobachtungen mit Neutronen und deren Aussagekraft im Bezug auf die Entwicklung des Universums. Es werden Experimente und neue Ergebnisse vorgestellt, die die Bedeutung dieses Forschungsfeldes demonstrieren, welches auf hochintensive Forschungsneutronenquellen angewiesen ist.
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<td>09:00</td>
<td>Plenary lecture: Neutron Depth Profiling: A Challenging New Method to <em>in situ</em> Monitor Lithium in Solid-State Li-Ion Batteries <em>R.A. Eichel</em></td>
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<td>09:35</td>
<td>Keynote lecture: Neutrons Scattering in Continuous Magnetic Fields up 26 T <em>Oleksandr Prokhnenko</em></td>
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Modern Science with Neutrons: Spanning the Arc from Hidden Quantum Order to Membrane Channels

Helmut Schober¹

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Keywords: neutron, quantum order, membrane channels

This plenary talk will give an overview of modern science with neutrons. We thank Prof. H. Schober for holding this talk on short notice, because Prof. R.A. Eichel could not come to the conference.
Neutrons Scattering in Continuous Magnetic Fields up 26 T

Olesandr Prokhnenko1, Peter Smeibidl1, Maciej Bartkowiak1, Wolf-Dieter Stein1, Norbert Stüßer1, Hans-Jürgen Bleif1, Sebastian Geriscker1, Robert Wahle1, Stephan Kempfer1, Jochen Heinrich1, Matthias Hoffmann1, Hartmut Ehmler1, Mark Bird2, Karel Prokes1, and Bella Lake1
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Keywords: neutron instrumentation, extreme sample environment, magnetic field

Recently, the Helmholtz-Zentrum Berlin (HZB) launched a unique high magnetic field facility for neutron scattering - HFM/EXED. It enables elastic neutron scattering experiments in continuous magnetic fields up to 26 T combined with temperatures down to 0.6 K1,2. The name HFM/EXED is derived from the acronym for two main components, the High Field Magnet and the Extreme Environment Diffractometer.

The HFM is a horizontal-field hybrid magnet, designed and constructed in collaboration with the National High Magnetic Field Laboratory (Tallahassee, USA). It opens not only the new opportunities for research in high fields but is itself at the forefront of development in magnet technology. Using a set of cable-in-conduit superconducting and resistive coils, a maximum field of 26.2 T can be reached for cooling power of 4 MW for the resistive part. The inner resistive coil has a 50 mm diameter room temperature bore with conical ends to allow neutron-scattering to detectors up to ±15° off the beam axis. Furthermore the magnet can be rotated by an additional 15° to access a larger reciprocal space region. The user samples can be cooled down to 0.6 K using a dedicated 3He-cryostat developed at the HZB.

The magnet is permanently installed on the EXED instrument. EXED is a multi-purpose time-of-flight (TOF) diffractometer optimized for operation with angular limitations imposed by the HFM. Due to the variable time resolution (from a few μs up to the ms-range) and the width of wavelength band (0.6-14.5 Å), the primary instrument is very flexible and adjustable to a particular problem. TOF technique combined with 15° magnet rotation provides a gapless coverage of Q-range from 0.1 up to 12 Å⁻¹ for diffraction experiments. The low-Q range can be extended beyond 10⁻² Å⁻¹ using a pin-hole TOF Small Angle Scattering mode implemented on the instrument. This year, in addition to the existing elastic capabilities, the instrument will be complimented by a direct TOF spectrometer mode. The latter will enable inelastic neutron scattering experiments over a limited Q-range < 1.8 Å⁻¹ with an energy resolution of a few percent and E_i < 25 meV.

In this talk the overview and capabilities of the HFM-EXED facility will be presented together with the first experimental results. Procedures for planning experiments and applying for the beamtime will be discussed.

References:
Session 4: Instrumentation II

Wednesday, Sept. 21, 2016, 10:30 - 12:00. Session Chair: Klaus Habicht
Location: Frederik-Paulsen-Hörsaal


10:45 - 11:00: The New Small-Angle Neutron Scattering Instrument SANS-1 at MLZ – Performance, Detector Characterization and First Results (ID 175) Sebastian Mühlbauer, Andreas Wilhelm, Lukas Karge, Andreas Ostermann, Ilario Defendi, Winfried Petry, Ralph Gilles, Andre Heinemann, Sebastian Busch, Andreas Schreyer

11:00 - 11:15: The Backscattering and Time-of-flight Spectrometer (BATS) Option for IN16B at ILL (ID 132) Markus Appel, Kristijan Kuhlmann, Bernhard Frick, Andreas Magerl

11:15 - 11:30: Upgrade of the Phase-Space Transform Chopper at SPHERES (ID 124) Michaela Zamponi, Marina Khaneft, Joachim Wuttke

11:30 - 11:45: Upgrade of TOF Spectrometer NEAT at Helmholtz-Zentrum Berlin – First Results (ID 228) Margarita Russina1, Gerrit Günther, Moritz-Caspar Schlegel, Veronika Sucha, Ramil Gainov

11:45 - 12:00: CAMEA — A Novel Multiplexing Analyzer for Neutron Spectroscopy (ID 198) Felix Groitl, Dieter Graf, Jonas Okkels Birk, Marton Markó, Marek Bartkowski, Uwe Filges, Raphael Müller, Christof Niedermayer, Christian Rüegg, Henrik M. Rønnow
Connecting MARIA with an MBE setup: first (quasi) in-situ neutron reflectivity measurements on thin films

Amir Syed Mohd\textsuperscript{1}, Sabine Pütter\textsuperscript{1}, Stefan Mattauch\textsuperscript{1}, Alexander Weber\textsuperscript{1}, Alexandros Koutsioubas\textsuperscript{1}, Harald Schneider\textsuperscript{1}, and Thomas Brückel\textsuperscript{1,2}

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Keywords: neutron, instrumentation, reflectometry, in-situ, thin films

MARIA is a dedicated instrument for neutron reflectometry of thin films. However, due to limited space a UHV system for thin film growth and in-situ measurements cannot be placed on-site. Hence, up to now ultra thin films which are sensitive to ambient air have been fabricated in the MBE system located in the thin film laboratory nearby and were covered by protecting cap layers in-situ. However these layers may change the physical properties of the sample, which is undesirable and prevents from further manipulation of the sample, e.g. growth of additional layers.

Recently, we have solved this problem by developing a handy mini UHV-chamber which is capable for both, sample transfer and quasi in-situ measurements at the neutron reflectivity instrument, respectively. Quasi in-situ polarized neutron reflectivity (PNR) measurements can be performed at room temperature in magnetic fields of up to 600 mT at MARIA.

Our solution consists of a DN CF-40 cube with two opposing sapphire windows for the neutron beam, a combined non evaporable getter and ion pump for keeping the vacuum, a wobble stick, which is needed for in-situ sample transfer and also serves as sample holder for samples of up to 1 cm\textsuperscript{2} and a valve for sample exchange by mounting the chamber on the MBE system. The pressure in the transfer chamber is kept in the $10^{-10}$ mbar range during transport and PNR measurement.

We present first polarized neutron reflectivity measurements on Co thin films at room temperature in a magnetic field of 300 mT in the Q-range up to 0.2 Å\textsuperscript{-1}.

Booking of the access to the MBE system as well as the transport chamber is possible via the MLZ user office system in combination with an application for a beam time at neutron instruments like MARIA.

This project is part of the nanoscience foundry and fine analysis project (NFFA, www.nffa.eu) and has received funding from the EU’s H2020 research and innovation programme under grant agreement N. 654360.
The New Small-Angle Neutron Scattering Instrument SANS-1 at MLZ – Performance, Detector Characterization and First Results

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A. Heinemann², S. Busch², A. Schreyer²
² German Engineering Materials Science Centre (GEMS) at Heinz Maier-Leibnitz Zentrum (MLZ), Helmholtz-Zentrum Geesthacht GmbH, Lichtenbergstr. 1, D-85748 Garching

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Keywords: SANS instrumentation, SANS detectors, properties of ³He tube array detectors

Abstract: A thorough characterization of the key features of the new small-angle neutron scattering instrument SANS-1 at MLZ, a joint project of Technische Universität München and Helmholtz Zentrum Geesthacht, is presented [1]. Measurements of the neutron beam profile, divergence and flux are given for various positions along the instrument including the sample position and agree well with Monte Carlo simulations of SANS-1 using the program McStas [2]. Secondly, the polarization option of SANS-1 is characterized for a broad wavelength band.

As a key feature of SANS-1 is the large accessible $Q$-range facilitated by the sideways movement of the detector, particular attention is paid to (i) the effects that arise due to large scattering angles on a position sensitive 2D detector made up from an array of single ³He tubes, where a standard $\cos^3$ solid angle correction is no longer applicable: These effects include tube shadowing, finite tube wall thickness and the anisotropic solid angle correction perpendicular and along the tube direction, respectively. In addition, (ii) dead time effects that arise due to high counting rates up to 2.5MHz at short detector distances are carefully characterized.

As final issue the performance of the instrument is characterized by a set of standard samples (diluted latex spheres and glassy carbon).

References:
The 'Backscattering And Time-of-flight Spectrometer' (BATS) Option for IN16B at ILL

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Keywords: Instrumentation, Neutron backscattering, Neutron time-of-flight, Spectroscopy

Since its commissioning in 2013, the backscattering spectrometer IN16B has proven its outstanding performance in serving the user community for high resolution experiments at the Institut Laue-Langevin (ILL) in Grenoble, France.[1] The most notable difference to its predecessor IN16 is the doubling of the maximum energy transfer range to ±31 μeV with the standard Si 111 monochromator & analyzers, and the increase of count rate by more than one order of magnitude. Low flux has been a long standing weakness of reactor based neutron backscattering, therefore past efforts in development mainly focused on improving neutron intensity. With the progress made in the IN16B project and the high flexibility provided by the instrument, new directions in instrumental development are pursued in the framework of a collaborative research grant („Verbundforschungsförderung“) by the German Federal Ministry for Education and Research (BMBF). On the one hand the possibility for significant improvement in energy resolution aiming at 50 neV is demonstrated in a GaAs 200 prototype spectrometer option,[2] while we focus here on the implementation of the Backscattering And Time-of-flight Spectrometer option „BATS“.

With the BATS option, IN16B can be transformed into an inverted time-of-flight spectrometer (see Fig. 1).[3] The maximum energy transfer is increased by an order of magnitude, while the energy resolution is slightly relaxed. With the standard Si 111 analyzer the expected energy resolution is tunable in the range of 2–9 μeV, the energy transfer range is ±250 μeV (movable) while the Q-range remains at 0.2–1.8 Å⁻¹. This option thus offers μeV-resolution at large Q in a range not accessible for direct geometry ToF instruments. A flexible pulse chopper system is to be installed in late 2016 consisting of two pairs of high speed counter-rotating choppers (diameter 750 mm, 19000 rpm) with multiple slits located 34 m upstream of the sample. The design allows to select from a set of different pulse lengths to optimize intensity vs. resolution for each experiment. Taking advantage of the continuous reactor source, a high pulse repetition rate mode (236 Hz) is available and allows for a fourfold increase in flux, albeit at the cost of reducing the energy transfer range to one third. Due to the extremely short chopper opening time of about 9 μs in order to achieve best resolution of 2 μeV, the chopper slits need to be smaller than the existing large guide cross section. To compensate for losses in flux we explore the possibilities of an adaptive focusing-defocusing guide system before and after the pulse chopper unit. Preliminary ray-tracing simulations are used in conjunction with a particle swarm optimization algorithm to refine a quasi-free guide shape for each chopper configuration separately, yielding a possible flux increase by a factor of 6 for the smallest chopper opening.

References:

Figure 1: Schematic layout of IN16B in (a) conventional backscattering and (b) BATS configuration.
Upgrade of the phase-space transform chopper at SPHERES

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Keywords: instrumentation

The Phase Space Transform (PST) chopper at the high resolution backscattering spectrometer SPHERES (SPectrometer for High Energy RESolution) has been upgraded. Besides its function as a duty-cycle chopper and primary beam deflector, the chopper achieves, by the motion of the mosaic graphite crystals on its circumference, a phase space transformation of the incoming wavelength spectrum, which enhances the usable flux at the monochromator.

The original three-wing PST chopper could not be operated at the optimal speed due to mechanical restrictions. It was operated only with 1/3 of the originally intended crystal speed of 300m/s. Newer simulations indicate an optimum speed for the phase space transformation around 250m/s. In order to use a larger fraction of the duty cycle, a one-hole chopper is preferred over the original three-hole geometry. Therefore a new more compact one-wing PST chopper has been designed. Together with the upgrade of the chopper also the graphite deflector crystals were replaced with ones of a higher reflectivity and mosaicity to further increase intensity.

Recently the new PST chopper was installed at the instrument and taken successfully into operation. It runs now with the desired frequency with a crystal speed of 225m/s close to the optimum velocity for the phase space transformation. Thanks to the increased velocity and the better deflector crystals, the intensity in most detectors has now been doubled. Beside the intensity increase of a factor 2, the signal-to-noise ratio for the large angle detectors have been improved by about 10%. The next step of reinforcing SPHERES is the optimization of the focusing neutron guide based on simulations which also consider the new PST chopper.
Upgrade of TOF spectrometer NEAT at Helmholtz-Zentrum Berlin – first results

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Keywords: neutron spectroscopy, instrumentation, dynamics

Abstract

Neutron time-of-flight spectrometer NEAT has a long history of successful application to the study dynamics and function. It is best suited to probe dynamic phenomena directly in space and time in the large time domain $10^{-13} - 10^{-10}$ s and on the length scale ranging from 0.05 to up to about 5 nm. To address the needs of user community in more powerful instrumental capabilities, a concept of full NEAT upgrade for substantial intensity increase was developed in addition to the pending improvements of the cold neutron source of BER II. The upgrade started in 2010 after a rigorous internal and external selection process. The new NEAT is currently in commissioning phase and here we report first results.

The advanced features of the new instrument include novel integrated guide-chopper system, which is optimized for both hard and soft matter studies and is several times more efficient in the use of the source power as world leader IN5 at ILL. Neutron guide delivers neutrons with flexible beam properties: either highly homogeneous beam with low divergence for single crystals studies or “hot-spot” neutrons suitable for small samples. Substantial increase of the detector angle coverage was achieved by using 412 $^3$He position sensitive detectors placed at 3 m from the sample and equipped by modern electronics and DAQ using event recording techniques. Combined with new instrumental and sample environmental capabilities the upgrade will allow NEAT to maintain at the best world class level and provide an outstanding experimental tool for broad range of research areas at BER II.
CAMEA — A novel multiplexing analyzer for neutron spectroscopy

Felix Groitl, Dieter Graf, Jonas Okkels Birk, Marton Markó, Marek Bartkowiak, Uwe Filges, Raphael Müller, Christof Niedermayer, Christian Rüegg, Henrik M. Rønnow

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Keywords: Neutron Instrumentation, Neutron Inelastic Scattering, Multiplexing, Neutron Spectrometer, Triple-Axis Spectrometer

The neutron spectrometer CAMEA (Continuous Angle Multiple Energy Analysis) will be installed as a new analyzer system on the cold-neutron triple-axis instrument RITA-2 at SINQ, PSI. CAMEA is optimized for efficiency in the horizontal scattering plane enabling detailed and rapid mapping of excitations. As a novelty the design employs a series of several sequential upward scattering analyzer arcs. Each arc is set to a different, fixed final energy scattering towards position sensitive detectors (PSDs). Thus, neutrons with different final energies are collected simultaneously over a large angular range.

For CAMEA in a single data-acquisition several entire constant-energy lines in the horizontal scattering plane are recorded for a quasi-continuous angular coverage of about 60°, whereby tremendous gains in data collection rates can be achieved. With a large combined coverage in energy and momentum, this will result in a very powerful and efficient spectrometer, which will be particularly suited for parametric studies under extreme conditions with restrictive sample environments (high magnetic field magnets or pressure cells) and for small samples of novel materials. We will present the analyzer concept, performance simulations, technical solutions and prototype verifications.

This project is a joint venture of the Laboratory for Neutron Scattering and Imaging (LNS, PSI), Laboratory for Developments and Methods (LDM, PSI) and the Laboratory for Quantum Magnetism (LQM, EPFL) financed by the R’Equip program of the Swiss National Science Foundation (SNF), EPFL and PSI.

References:

M3_1: Minisymposium “Materials characterisation”, part 1

Wednesday, Sept. 21, 2016, 10:30 - 12:00. Session Chair: Michael Hofmann
Location: Hörsaal C

10:30 - 10:55: Orientation Dependent Variant Selection Induced by Thermal-mechanic Treatments in a Polycrystalline Ni-Mn-Ga Alloy Studied by Neutron Diffraction (ID 271) Yudong Zhang


11:10 - 11:25: Stabilization of Quantum Dots Characterized by In Situ Small Angle Scattering (ID 208) Torben Schindler, Tilo Schmutzler, Martin Schmiele, Wei Lin, Doris Segets, Wolfgang Peukert, Tobias Unruh


Orientation Dependent Variant Selection Induced by Thermal-mechanic Treatments in a Polycrystalline Ni-Mn-Ga Alloy Studied by Neutron Diffraction

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Keywords: Ni-Mn-Ga alloys, Martensitic transformation, Neutron diffraction, Variant selection, Thermal-mechanical treatment

The Ni-Mn-Ga 5M martensite presents a considerable magnetic field induced strain (MFIS) up to ~6% with a relatively small twinning stress (~0.1MPa). To obtain large MFIS, the microstructure modification through external field treatment to reorganize variant configuration is supposed to be an essential prerequisite. As the lattice deformation of the martensitic transformation is anisotropic, imposition of a unidirectional constraint could promote the formation of favourable variants but prevent unfavourable ones. Therefore, the loading direction determine the formation of the final martensitic microstructure. Deep insights into the variant redistribution feature caused by thermo-mechanical treatment (i.e. application of external load during the martensitic transformation) as well as its correlation to the loading direction should not only enrich the martensitic transformation crystallography, but also offer some necessary guidelines for the design and optimization of the external training process.

In this work, the influences of uniaxial compressive load on martensitic transformation from austenite to 5M martensite were studied in a directionally solidified Ni-Mn-Ga polycrystalline alloy with coexisting <0 0 1>ᴬ and <1 1 0>ᴬ preferred orientations parallel to the solidification direction (SD). Based on the neutron diffraction, the direct evidence on the variant redistribution induced by the thermal-mechanical treatment was presented and the selection of preferential variants was found to be strongly dependent on the austenite orientation. For the austenite with a <0 0 1>ᴬ preferred orientation, the compressive loading direction (LD) along the SD can promote the formation of preferred variants with {0 2 0} captcha(3) SD (LD). On the other hand, for the austenite with a <1 1 0>ᴬ preferred orientation, the variants with {1 2 5} captcha(5) SD (LD) are more favorable after the thermal-mechanical treatment. Such variant selection is originated from the accommodation between the anisotropic lattice distortion in martensitic transformation and the external constraint. The present investigations may offer some fundamental information on variant selection subject to external stress field and the necessary guidelines for microstructure optimization of polycrystalline Ni-Mn-Ga alloys through external field training.
TaC Precipitate Stability in Co-Re Base Superalloys, Investigated with Neutrons

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Keywords: Superalloy; Small-angle neutron scattering; Neutron diffraction; phase transformation

It is becoming increasingly clear that new materials - operating at substantially higher temperatures than current Ni-base superalloys - are needed for future gas turbines. High melting Co-Re-Cr based alloys strengthened by carbides, particularly MC type, show promise [1]. A stable dispersion of fine TaC precipitates provides the high temperature creep strength. Additionally, Cr - which is mainly added to improve oxidation resistance - also stabilizes lamellar M23C6 type Cr carbide. It is well known that pure Co undergoes an allotropic phase transformation from ε (hcp) phase at RT to γ (fcc) phase at high temperatures and this was also recorded in various Co-Re alloys in in-situ neutron diffraction measurements [2]. The transformation temperature is strongly affected by alloying addition and interferes with TaC precipitation. Re, a hcp stabilizer, increases the transformation temperature and Ta as fcc stabilizer decreases it. Therefore, the coherency and stability of the TaC precipitates are affected by the Co-matrix transformation. As it is apparent from the binary Ta-C phase diagram that the TaC phase exists over a wide composition range (C/Ta = 0.5 – 1), the stability of TaC was studied in a series of Co-Re-Ta-C alloys with varying C/Ta ratio.

In this contribution, the influence of the C/Ta ratio on size, volume fraction and high temperature stability of fine TaC is presented, studied by means of in-situ small-angle neutron scattering (SANS) and in-situ neutron diffraction. It is shown that size and volume fraction strongly depend on this ratio, and the fine TaC (< 100 nm) are present in the Co-Re matrix up to temperatures of T = 1200 °C (Figure 1) [3]. In addition, the influence of allotropic Co-Re matrix transformation hcp ↔ fcc on precipitates is discussed.

References:
Stabilization of ZnO Quantum Dots Characterized by In Situ Small Angle Scattering

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Keywords: neutrons, X-rays, small angle scattering, nanomaterials characterization

Semiconductor nanoparticles are of special interest in research as their properties can be adjusted due to the quantum size effect which allows many applications in different electronic devices and thin film solar cells. To design stable and well-defined nanoparticles knowledge about the growth processes and the influence of stabilizers is crucial. Standard characterization techniques can determine the size of the nanoparticle core (SAXS, AUC, UV/Vis, TEM) but lack in characterizing the stabilization shell. Using SANS information regarding the organic stabilizer can be achieved, but it is the combination of in situ SANS and SAXS, which allows to obtain a deeper understanding of the stabilization due to the different contrasts of the two in situ methods toward quantum dot core and organic shell.

By the simultaneous analysis of SANS and SAXS scattering patterns of the same sample on absolute scale we were able to observe the stabilizing shell of ZnO nanoparticles in their native solution for the first time (Figure 1A+C). Furthermore, we studied the effects of purification of the ZnO nanoparticles and its influence on the shell using this method. In addition the results from ligand exchange reactions with a catecholic stabilizer will be presented. (cf. Figure 1B+D).

Figure 1: SAXS (green open squares) and SANS (blue open circles) data and fit curves of the simultaneous fit for ZnO nanoparticles A) in the native dispersion and B) after ligand exchange with a catecholic stabilizer. The dotted lines in A) show the influence on the fit curve, if the shell is neglected. C) and D) show the stabilization of the ZnO nanoparticles after synthesis and after ligand exchange, respectively.

Small-Angle Neutron Scattering of Lithium-Ion-Batteries

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Keywords: small-angle neutron scattering, Li-ion battery, graphite anode, in-operando

Small-angle neutron scattering is a versatile technique to study inhomogeneities in the nanoscale region. Objects such as precipitates or pores can be studied in respect to size distribution and volume fraction. Also, phase changes from an entire macroscopic sample and phenomena such as the evolution of a shell surface layer can be measured. We applied this technique to follow the lithiation of battery materials, i.e. in our case the lithiation of graphite particles as the standard anode material in today’s Li-ion batteries.

One can show that the macroscopic differential cross section and thus also the scattered intensity $I(q)$, which is a function of scattering angle or wave vector $q$, are proportional to the squared difference in scattering length density $\Delta \rho^2$ of the observed phases. But coherent scattering can only occur from within a certain coherent volume of a few hundred nanometers which adds local information. We integrate $I(q)$ over a wide $q$-range to get a measure of the total scattering, very similar to the well-known Porod-invariant. In earlier work¹, we have shown that one can indeed follow the lithiation of a graphite particle from outside to inside in a full cell versus LiNi$_{1/3}$Mn$_{1/3}$Co$_{1/3}$O$_2$. Also versus Li in a half cell (shown in figure 1), one can see the lithiation via the two major phases LiC$_{12}$ and LiC$_{6}$ which show up in the potential curve. Because only $\Delta \rho^2$-contrast from these phases to the surrounding electrolyte matters and it only contributes to scattering from within the coherent shell, the integrated scattering intensity varies at the beginning of the lithiation of a new phase (a, in Fig. 1) but is constant during the lithiation of the inner particle (b). We have analyzed the small-angle neutron scattering data of graphite in several half and full cells under different conditions and will present theoretical basics, practical considerations, results and future developments of this work, also in comparison to other techniques.

References:

\(\gamma/\gamma'\) lattice misfit and the evolution of \(\gamma'\) precipitates of Co-base superalloys studied by neutron diffraction and small-angle neutron scattering

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Keywords: diffraction, small-angle scattering, Cobalt, superalloys, microstructure

Abstract: The discovery of the \(\gamma'\) phase Co\(_3\)(Al,W) with L\(_{12}\) crystal structure one decade ago has led to the development of novel Co-base superalloys with attractive properties for high temperature applications. High-strength, creep and oxidation resistant Co-base superalloys consisting of a high volume fraction of coherent \(\gamma'\) precipitates within an fcc \(\gamma\) solid solution have been designed by the authors\(^1\). Despite their similarity to conventional Ni-base superalloys several differences such as the sign of the lattice misfit between \(\gamma\) and \(\gamma'\) phase exist.

In this work neutron diffraction (ND) and small-angle neutron scattering (SANS) methods at MLZ were used to characterize in detail the phases of this new class of high temperature materials.

ND measurements were performed to study how the \(\gamma/\gamma'\) lattice misfit changes with temperature and how alloying elements such as Ta and Cr influence the lattice misfit\(^2\). Special emphasis was placed on the role of the base element Co. ND investigations on model Ni/Co-9Al-8W-8Cr superalloys show for example, that the lattice misfit changes from negative to positive values if Ni is successively substituted by Co\(^3\). This is caused by the continuous change of the partitioning behavior of Al and W when Ni is replaced systematically by Co as found by atom probe microscopy investigations.

Multi-step heat treatments have to be developed in order to obtain a multimodal \(\gamma/\gamma'\) microstructure for achieving a high strength. SANS investigations on the evolution of the strengthening \(\gamma'\) phase at different aging temperatures revealed how the secondary and tertiary \(\gamma'\) precipitates grow and how to optimize the aging treatment.

References:
**Structural Investigation of Single and Interacting Soft Interfaces by Neutron Reflectometry**

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Keywords: biological interfaces, neutron reflectometry, interfacial forces, polymer brushes

Soft interfaces constituted by molecular assemblies in two dimensions play important roles in numerous technological applications and are major components of all biological matter, for example in the form of biomembranes. The understanding of important biological or technological processes involving single (isolated) or interacting soft interfaces typically relies on detailed structural insight. Neutron reflectometry (NR) is uniquely suited for the structural investigation of soft and dynamic interfaces at molecular length scales. Here, this great potential will be illustrated exemplarily for surfaces rendered with hydrated layers of linear macromolecules: (i) Surfaces functionalized with polymer brushes in order to prevent protein adsorption and (ii) the lipopolysaccharide surfaces of Gram-negative bacteria.
### Session 5: Soft Matter / Biology / Medicine

**Wednesday, Sept. 21, 2016, 13:40 - 15:10. Session Chair: Thomas Hellweg**

**Location:** Frederik-Paulsen-Hörsaal

Igor Krasnov, Tilo Seydel, Martin Müller

13:55 - 14:10: **Swelling Kinetics of N-n-propylacrylamide Based Microgels upon Periodic Pressure Jumps Investigated by Time-resolved Small Angle Neutron Scattering** *(ID 242)*  
Oliver Wrede, Yvonne Pütz, Stefan Lülsdorf, Diana Zauser, Yvonne Hertle, Ralf Schweins, Thomas Sottmann, Thomas Hellweg

14:10 - 14:25: **Thinning of DMPC-Bilayer Induced by the Incorporation of the Saponin Escin** *(ID 231)*  
Carina Dargel, Ramsia Sreij, Jacques Jestin, Thomas Hellweg

14:25 - 14:40: **Influence of Membrane Proteins on Lipid Dynamics and Membrane Structure** *(ID 236)*  
Lisa Lautner, Tilo Seydel, Tobias Unruh

14:40 - 14:55: **Evidence for Hydrated Channels and Connected Water Clusters in Proton Conductive Membranes Based on Sulfonated Syndiotactic Polystyrene** *(ID 144)*  
Maria Maddalena Schiavone, Marie-Sousai Appavou, Aurel Radulescu, Oreste Tarallo, Rocco Di Girolamo, Lucia Caporaso, Zsolt Revay, Dieter Richter

14:55 - 15:10: **Pharmaceutical and Medical Nanoparticles – Drug Interaction with Gastro-Intestinal Nanosystems and Target-Nanoparticles for Cancer Therapy** *(ID 255)*  
Thomas Nawroth, Pooneh Khoshakhlagh, Lidija Krebs, Peter Langguth, Gunther Goerigk, Noemi Szekely, Ralf Schweins
Fractional dynamics in *Bombyx mori* silk studied by *in situ* QENS scattering experiments.

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Keywords: neutron, biomaterials, structure, dynamics

Structural relaxations in humid silk fibers exposed to tensile stress have been observed to take place on a very wide range of time scales from a few milliseconds to several hours. The time-dependence of the measured tensile force following a quasi-instantaneously applied external strain on the fibers can be understood in terms of a fractional viscoelastic relaxation function introducing memory effects by which the mechanical state of a fiber depends on its tensile history.¹ An analog fractional relaxation also gives rise to the subdiffusion observed on picosecond time scales, which governs the mobility of the amorphous polymer chains and adsorbed water on the molecular level. The reduction of the subdiffusive memory effect in stretched fibers compared to native fibers is consistent with the higher order of the polymers in the stretched state.

References:
Swelling kinetics of N-n-propylacrylamide based microgels upon periodic pressure jumps investigated by time-resolved Small Angle Neutron Scattering

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Keywords: microgels, SANS, polymer dynamics

Temperature responsive microgels are particles in the size range between 100 nm and 1 \( \mu \)m, that undergo a reversible microphase separation at a specific temperature, the so called Volume phase transition temperature (VPTT). Structure, size and phase transition properties can be controlled by the synthetic conditions, e. g. crosslinker content, surfactant concentration or different monomers/comonomers. This makes them outstanding candidates for various applications, such as drug delivery, catalytic particle carriers or nanoactuators. A precise understanding of the underlying processes is necessary to tune the network properties precisely for the different applications. Therefore, we investigated the swelling and deswelling kinetics of N-n-propylacrylamide (NNPAM) based microgels upon periodic pressure jumps across the phase transition with different crosslinker contents by time-resolved SANS. Preceding DLS measurements showed, that the phase transition shifts towards higher temperatures with increasing pressure, i.e. a swelling of the particles is induced upon compression. Due to the sharp phase transition, NNPAM based microgels are especially suited for this experiments, as both the swollen as the collapsed state can be accessed at the same temperature by applying different pressures. Our findings show, that the time scale of the kinetics changes with cross-linker content by more than one order of magnitude, which confirms the important role of the network morphology. Besides the influence of the cross linker, the kinetics of the swelling is considerably faster than the deswelling process.

Figure 1: SANS measurements of the deswelling/swelling kinetics of a N-n-propylacrylamide based microgel by periodic pressure jumps across the phase transition.
Thinning of DMPC-Bilayer Induced by the Incorporation of the Saponin Escin

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Keywords: unilamellar vesicle (ULV), DMPC, saponin, escin, small angle neutron scattering (SANS)

The interaction of biological membranes with different agents, e.g. nonsteroidal anti-inflammatory drugs (NSAIDs) is often studied on model membranes in the form of unilamellar vesicles (ULVs) composed of the phospholipid 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC). Because of its amphiphilic character DMPC molecules can arrange to lipid bilayers with a hydrophobic interior, into which small hydrophobic molecules can incorporate and thereby change the properties of the lipid membrane. An example is the NSAID ibuprofen, for which an incorporation into the DMPC-membrane and thereby a significant thinning of the headgroup region was observed by small angle neutron scattering (SANS) methods by Boggara et al. Moreover, the incorporation of ibuprofen, or like in living organisms of cholesterol, decreases the main phase transition temperature from gel to fluid phase ($T_m$ for pure DMPC vesicles ~23.6 °C). Another class of agents, assumed to at least strongly interact with lipid membranes are the amphiphilic saponins, which are built of a hydrophobic steroidic or triterpenic backbone with a varying number of hydrophilic sugar chains. Saponins are natural products known for their cholesterol lowering effect due to complex formation with cholesterol.

For investigating in detail the interaction of saponins itself with (biological) membranes, a deductive study on ULVs composed of DMPC and varying amount of the pure saponin escin was implemented. Thereby differential scanning calorimetry (DSC) and photon correlation spectroscopy (PCS) measurements yield that below a limiting amount ($\chi_{\text{limit}}$~1.5 mol%) escin is only attached to the membrane surface but incorporates into the membrane above this amount. Besides lowering of $T_m$ escin molecules show raft formation similar to cholesterol within the membrane, which is accompanied by aggregation effects, as deduced from turbidity measurements and wide angle X-ray scattering (WAXS). By SANS and analysis via a modified Kratky-Porod (MKP) plot a thinning effect of the membrane caused by the escin incorporation was proofed (cf. Fig. 1). Further the distribution of the escin molecules in the membrane will be investigated by contrast variation methods.

References:
Influence of membrane proteins on lipid dynamics and membrane structure

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Proteins and lipids constitute a major part of biological cell membranes. The lipid-protein interactions effect a variety of important cell function as i.a. the regulation of molecular and signal transport which are strongly coupled to structure, composition and dynamics of the membrane.

Neutron scattering is a powerful tool to probe structure and dynamics of biological membrane mimics on an atomistic scale [1,2]. This allows studies on e.g. the diffusion of lipids, the undulation motions of membranes or also the mutual interactions of the membrane constituents. Using the backscattering spectrometer IN16B (ILL, Grenoble, France) quasielastic neutron scattering (QENS) experiments were performed on phospholipid 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC) vesicles at 310 K with and without the transmembrane sequence of the transferrin receptor protein to study the influence of the protein on the lipid dynamics. The experiments indicate a restricted lipid mobility for the lipid-protein vesicles compared to the pure lipid vesicles. First results of these measurements and the analysis of the fundamental question how far the influence of the proteins reaches on the surrounding lipids will be presented here.

Besides dynamics, the proteins also affect the membrane structure for that reason it is important to obtain also detailed understanding about protein position and binding at the lipid bilayer. Therefore, a combined SAXS/SANS study were performed at the small-angle neutron spectrometer D11 (ILL, Grenoble, France) and the in-house x-ray scattering instrument VAXSTER. Initial results of these studies will be presented as well.

Evidence for Hydrated Channels and Connected Water Clusters in Proton Conductive Membranes Based on Sulfonated Syndiotactic Polystyrene

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Keywords: sulfonated syndiotactic polystyrene, clathrate, SANS.

Syndiotactic polystyrene (sPS) is able to form different kinds of co-crystalline phases with guest molecules of various size, shape and property. Several advanced materials have been already produced starting from sPS co-crystalline films (like for example fluorescent, photoreactive, chiral and paramagnetic materials). In particular, sulfonated sPS (sSPS) can be used as proton-conductive membrane for fuel cells, as it presents high proton conductivity (comparable with Nafion), it shows a high chemical and thermo-mechanical stability and it has a low cost. The morphology of different sPS clathrates and the structural features of sSPS upon hydration can be more thoroughly understood by combining X-rays scattering, FT-IR and TEM with SANS. By exploiting the neutron contrast variation between various hydrogenated and deuterated components of sPS and sSPS clathrates, additional and unique information about the distribution of guest molecules in the crystalline and amorphous regions and about the hydrated domains were obtained. Moreover, the SANS investigation of in-situ water absorption-desorption process in these membranes using a humidity chamber that enables to choose constant relative humidity RH over a wide range (between 5% and 95%) emphasizes that the hydration-drying processes involve both the inter-lamellar amorphous space and bulk amorphous. Moreover, the stretching of films leads to occurrence and distribution of scattering features from typical morphologies on specific directions and sectors of detection plan, which enables an unambiguous characterization of such complex polymeric systems. A complete SANS investigation on sPS samples, starting from their crystallization with guest molecules through sulfonation process and followed by subsequent hydration, performed at SANS diffractometer KWS-2 of MLZ will be presented. The SANS results were complemented with WAXS, FTIR and TEM observations. This experimental analysis has highlighted that the morphology of these polymeric films is characterized by hydrated channels in the amorphous phase which are aligned along the stretching direction and alternate to staples of crystalline lamellae.

References:
[4]. F. Kaneko et al., Polymer, 54, 3145 (2013); DOI:10.1016/j.polymer.2013.04.050
[5]. F. Kaneko et al., Chemistry Letters, 44, 497 (2015); http://doi.org/10.1246/cl.141179
Pharmaceutical and Medical Nanoparticles – Drug Interaction with Gastro-Intestinal Nanosystems and Target-Nanoparticles for Cancer Therapy

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Keywords: SANS, nanoparticles, structure, dynamics, medical application

Nanoparticles play a central role in two branches of pharmaceutical and medical applications: a) transient nanoparticles serve as intermediate carriers of vitamins and drug from oral applied medicine in the human gastro-intestinal system, and b) nanoparticles can serve as drug carriers, which can be personalized by surface modification for a cell specific recognition and uptake, e.g. in cancer therapy. Those nanocarriers can be micelles, liposomes and polymer nanoparticles, which have to be structurally correct, biocompatible and bio-degradable. Thus the development of pharmaceutical nanoparticles needs a combination of pharmaceutical, medical and structure research methods in a feed-back process. The components of the complex nanoparticles from several materials are distinguished by neutron small angle scattering SANS with deuterium-contrast variation, dynamic processes during application and protein binding are studied by time resolved SANS, a localization of heavy metals for radiotherapy requires energy dependent x-ray scattering ASAXS, while the wide size scale requires a parallel investigation with dynamic light scattering DLS (PCS).

Transient nanoparticles from bile, food and drug formulation are investigated in a simulated gastro-intestinal system (device) by SANS and DLS. The flow-through setup at KWS2 at the FRM-II reactor models the human duodenum by an elongated quartz cell (300 mm), which is fed through a mixer and pumps by simulated bile, fluid from a stomach model and drug formulation. Fast processes are studied by the stopped flow technique. Hydrophobic drugs include in the sequence of micelles and liposome species which are sequentially forming. We found that the cholesterol content of the bile changes this drug solubilization and kinetics [1], which shall utilized in new drug formulations.

The medical nanoparticles for parenteral application, e.g. cancer therapy, are liposomes as fast development system and stable biodegradable polymer nanoparticles, which can be loaded with heavy metals for radiotherapy with photons or neutrons, both inactivating cells by secondary radiation products as free radicals. Both systems, and artificial intestinal nanoparticles, are functionalized by surface modification with targeting excipients [2], bearing a terminal charge, ligand, cofactor or protein corona. This shall serve as key for a personal medicine.

References:
M3_2: Minisymposium “Materials characterisation”, part 2

Wednesday, Sept. 21, 2016, 13:40 - 15:10. Session Chair: Peter Staron
Location: Hörsaal C

13:40 - 14:05: Determining Fundamental Properties From Diffraction: Electric Field Induced Strain And Piezoelectric Coefficient (ID 126)
Manuel Hinterstein, Markus Hoelzel, Andrew Studer, Michael Hoffmann

14:05 - 14:20: Effect of Base Metal Texture on Microstructure, Tensile Properties and Residual Stresses of Laser-Welded Titanium Joints (ID 113)
Emad Maawad, Weimin Gan, Michael Hofmann, Volker Ventzke, Stefan Riekehr, Heinz-Günter Brokmeier, Nikolai Kashaev, Martin Müller


14:35 - 14:50: Investigation of Hydrogen Storage Tanks using Neutron Imaging (ID 195) Stefan Börries, P. Klaus Pranzas, Oliver Metz, Thomas Bücherl, Michael Schulz, Jose M. Bellosta von Colbe, Martin Dornheim, Thomas Klassen, Andreas Schreyer, Martin Müller
Determining fundamental properties from diffraction: Electric field induced strain and piezoelectric coefficient

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Keywords: neutron, piezoceramics, texture, electric field

Piezoelectric ceramics exhibit the remarkable property to couple elastic strain and polarization under the influence of an applied electric field. Among the various types of piezoelectric devices, especially actuators rely on high electric fields to generate high strains and forces. Prominent examples for actuators are multilayer stack actuators used for nanopositioning or in modern combustion engines for automobiles to control injection cycles. The two most important characteristics of this class of materials are macroscopic strain and piezoelectric coefficient. Despite extensive studies and elaborated measurement techniques, the correlation between macroscopic strain and structural response is still not fully understood.

Most of the relevant systems found up to now are compositions close to phase boundaries linking highly correlated phases. This results in major challenges for structural analyses due to overlapping reflections. Apart from the well-known field induced structural responses such as domain switching and the converse piezoelectric effect we recently identified field induced phase transitions in different systems as an additional poling mechanism \cite{1,2}. In order to resolve all three involved poling mechanisms within only one experiment we developed a structural analysis technique with in situ X-ray and neutron powder diffraction data \cite{3}, using the program MAUD \cite{4}. The results not only separately reveal the contributions of each poling mechanism to the macroscopic strain, but also different behaviours of the individual phases. The calculation of the elastic strain perfectly matches the macroscopic observations, confirming the accuracy of the applied models. Since this method yields fundamental information such as the crystal structure as a function of applied electric field, we were able to calculate the piezoelectric coefficient for the individual phases based on information on the atomic scale. In this contribution we present the latest research on the elucidation of strain mechanisms and fundamental properties in piezoceramics.

\begin{thebibliography}{9}
\end{thebibliography}
Effect of Base Metal Texture on Microstructure, Tensile Properties and Residual Stresses of Laser-Welded Titanium Joints

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Keywords: laser welding, crystallographic texture, microstructure, residual stress, titanium

Single crystals show in general anisotropic behavior in case of 4th rank tensor properties (e.g. Young’s modulus and yield locus) and in case of 2nd rank tensor properties for all non-cubic materials (e.g. thermal expansion, thermal conductivity and compressibility) [1]. Therefore, the given crystallographic texture affects the properties of polycrystalline material. This can have consequences for the microstructure, mechanical properties and residual stress state of welded joints. In the present work, this influence has been investigated for the case of laser-welded titanium sheet material.

Commercially pure rolled titanium sheets were laser welded using an 8 kW fiber laser at 4 kW laser power with the weld line along rolling direction (RD), transverse direction (TD) and 45° to RD. The three-dimensional strain profile and the local texture around the weld were measured by neutron diffraction at the materials science diffractometer STRESS-SPEC [2] of the Heinz Maier-Leibnitz Zentrum (MLZ). Furthermore, grain orientation within the base material, the heat-affected zone and the fusion zone was investigated using the electron back-scatter diffraction (EBSD) technique. Tensile testing was performed at room temperature on flat tensile samples cut transversely across the weld.

The results revealed that the tensile properties differ in various welded sheets having different volume fractions of grains with a certain orientation with respect to the welding direction, while no pronounced texture effect on the residual stress state was observed. In addition, finer grains in the heat affected zone were developed on a joint side where the RD is parallel to the welding direction [3].

Advanced Material Characterization using Diffraction Contrast in Neutron Imaging

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Keywords: imaging, engineering materials, tomography, phase transformations, texture

Neutron imaging has experienced tremendous progress over the last decade, due to development of detector technology and by exploiting new contrast mechanisms, enabling phase contrast-, dark field contrast-, polarized neutron-, and neutron resonance absorption- imaging. Utilizing diffraction contrast offers the possibility to explore the microstructure in the bulk (cm range) of crystalline materials in 2D and 3D. This presentation will briefly outline the current status of transmission based neutron imaging at reactor and spallation sources. It will focus on vivid examples, showing local variations of strain induced phase transformation and texture evolution.

The new pulsed neutron sources unlock further potential in neutron imaging. Ongoing method development includes full pattern refinement of spectral transmission data as well as complementing transmission with diffraction data recording, paving the way towards 3D-neutron diffraction for exploring information on the individual crystalline grain level.

![Figure 1. Examples of Diffraction Contrast in Neutron Imaging: 3D reconstructed phase fractions inside a Tensile and a Torsion sample, where large plastic deformation leads to the formation of martensite.](image)

References:


Investigation of Hydrogen Storage Tanks using Neutron Imaging

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Keywords: neutron, imaging, hydrogen storage, metal hydrides

Hydrogen is a promising energy carrier for the future, both for mobile and stationary applications. It can be stored safely and reversibly at high volumetric densities in hydrogen storage tanks filled with light metal hydrides.

Due to the sensitivity of neutrons towards hydrogen, Neutron Imaging (NI) is the ideal technique for investigations addressing the hydrogenation behaviour of metal hydride materials inside a storage tank under operating conditions. While low and medium temperature hydrides [e.g. 1,2] have been already studied, first-time NI measurements of a hydrogen storage tank filled with the high temperature complex hydride LiBH₄-MgH₂ at the instruments NECTAR and ANTARES at FRM II have been performed. Combining the cold and fission neutron spectra of both instruments and using a new method for the quantitative investigation of neutron imaging data [3,4] it is possible perform a comprehensive study of the hydrogen distribution and material structure. The analyses possibilities and results provided by NI heavily impact the design and tailoring of future metal hydride beds and storage tanks towards application.

References

M4_1: Minisymposium “Neutron spectroscopy for dynamics in confinement”, part 1

Wednesday, Sept. 21, 2016, 13:40 - 15:10. Session Chair: Bernhard Frick
Location: Hörsaal D


13:55 - 14:15: Dynamics of Liquids in Nanoporous Silicon and Carbon as Studied by Quasi-Elastic Neutron Scattering (ID 190) Mark Busch, Tommy Hofmann, Patrick Huber

14:15 - 14:35: Neutron- and dielectric spectroscopy - taking advantage of the strengths of each technique in confinement studies: Vibrational and Molecular Dynamics of Nanoconfined Liquid Crystals (ID 191) Andreas Schönhals, W. Lohstroh, M. Zamponi, B. Frick, R. Zorn

14:35 - 14:55: Dynamics of Polymer Melt in Nanoconfinement (ID 181) Margarita Krutyeva, Michael Monkenbusch, Stefano Pasini, Jon Maiz, Carmen Mijangos, Andreas Wischnewski, Deter Richter

Review of neutron spectroscopy for studies of the dynamics in confinement

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Keywords: neutron, confinement, dynamics, soft matter, glass, polymer

The dynamics of liquids and polymers confined in nanoscopically small (1...10 nm) pores, tubes or lamellae has become a field of increasing interest in experimental physics. In this introductory talk three exemplary studies using inelastic/quasielastic neutron scattering [1] will be presented.

1. Quasielastic neutron scattering is used to study the α relaxation in glassforming liquids in confinement [2]. Neutron scattering is a suitable method to study such composite materials because the scattering of the liquid component can be emphasized by proper choice of isotopes. These studies may contribute to the understanding of the glass transition which is still one of the unsolved problems in solid state physics. Especially, the question of a cooperativity length scale may be addressed. These experiments usually require a large dynamical range which can be accessed by combination of backscattering and time-of-flight spectroscopy in the time domain.

2. Incoherent inelastic neutron scattering gives direct access to the vibrational density of states. In amorphous materials the VDOS shows a characteristic enhancement over the value expected from sound waves. The reason for the deviation from the crystalline spectrum is still heavily discussed. Studies of this phenomenon, the so-called boson peak, in confinement provide information on its origin [3].

3. Neutron spin-echo spectroscopy is a standard tool to investigate the chain dynamics in polymers (Rouse, reptation) and can also be applied for polymers in confinement. In the ideal case contrast matching should be able to suppress residual small-angle scattering from the confining pore structure. Although for subtle statistical reasons contrast matching does not work as well for polymers as it does for low-molecular materials [4], valuable information can be obtained.

References:
Dynamics of Liquids in Nanoporous Silicon and Carbon as Studied by Quasi-Elastic Neutron Scattering

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quasi-elastic neutron scattering, ionic liquids, hydrocarbons, mesoporous silicon, mesoporous carbon.

Spatial confinement in nanoporous media affects the structure, thermodynamics and mobility of molecular soft matter often markedly. Here, we present incoherent quasi-elastic neutron scattering measurements in a wave vector transfer range from 0.4 Å⁻¹ to 1.6 Å⁻¹ on liquid n-hexane confined in cylindrical, parallel-aligned nanochannels of 6 nm mean diameter and 260 µm length in monolithic, mesoporous silicon. They are complemented with, and compared to, measurements on the bulk system in a temperature range from 50 K to 250 K. The time-of-flight spectra of the bulk liquid (BL) can be modeled by microscopic translational as well as fast localized rotational, thermally excited, stochastic motions of the molecules. In the nano-confined state of the liquid, which was prepared by vapor condensation, we find two molecular populations with distinct dynamics, a fraction which is immobile on the time scale of 1 ps to 100 ps probed in our experiments and a second component with a self-diffusion dynamics slightly slower than observed for the bulk liquid. No hints of an anisotropy of the translational diffusion with regard to the orientation of the channels’ long axes have been found. The immobile fraction amounts to about 5% at 250 K, gradually increases upon cooling and exhibits an abrupt increase at 160 K (20 K below bulk crystallization), which indicates pore freezing.

Moreover, we present quasi-elastic neutron backscattering experiments on an ionic liquid (1-N-buty1pyridinium bis- (trifluoromethyl)sulfonylimide [BuPy] [Tf2N]) imbibed in carbide-derived mesoporous carbon samples. The analysis of quasi-elastic spectra enables us to study the thermally-activated molecular mobility of the spatially nanoconfined ions as a function of temperature and pore diameter. Foregone elastic and inelastic fixed window scans while the temperature is varied between 2 K and 350 K were particularly helpful to select the temperature for the measurement of the quasi-elastic spectra and provide global insights into the activation of molecular motions in the confined state.

References:
Neutron- and dielectric spectroscopy - taking advantage of the strengths of each technique in confinement studies:
Vibrational and Molecular Dynamics of Nanoconfined Liquid Crystals

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Keywords: Neutron Spectroscopy, Dielectric Spectroscopy, Confinement, Liquid Crystals

The effect of a nanometre confinement on the molecular dynamics of liquid crystals was studied by broadband dielectric and neutron spectroscopy. As confining host the molecular sieve Al-MCM-41 (Si/Al=60) with a mean pore diameter of 3 nm was selected. As guest the liquid crystals 8CB and E7 were chosen where the latter does not crystallize but undergoes a glass transition forming a nematic glass.

Dielectric spectroscopy shows that for the confined systems one relaxation process is observed. Its characteristic relaxation time is much lower compared to that of the bulk. No signature of the phase transition characteristics of the bulk liquid crystal is detected. The temperature dependence of the relaxation time of this relaxation process has to be described by the Vogel-Fulcher-Tammann equation and thus shows similarities to glassy dynamics.

The vibrational density of states measured by neutron spectroscopy (IN6, ILL; TOFTOF, MLZ) shows excess contributions with respect to the Debye density of states (boson peak). For the confined systems the low frequency contributions of the boson peak are suppressed, an effect which is also found for conventional glass forming systems.

In addition elastic scans were carried out at the backscattering spectrometer (BS) IN10 at ILL. Such measurements provide an overview about the temperature dependence of the microscopic dynamics. Assuming a Gaussian form for the elastically scattered intensities the effective mean square displacement was calculated. For the nanoconfined samples the mean square displacement is strongly reduced in comparison to the bulk. Also these measurements show the signature of a glass transition indicated by a change in the temperature dependence of the mean square displacement. For confined E7 the extracted glass transition temperature is shifted by more than 20 K to higher temperatures.

Broadband inelastic neutron scattering was carried out by combining time-of-flight (IN6, ILL; TOFTOF, MLZ) and backscattering (IN10, ILL; SPHERES, MLZ) experiments in the time domain. The time dependence of the incoherent intermediate scattering function $S_{\text{inc}}(q,t)$ shows two relaxation processes which were quantitatively analyzed by fitting the KWW function to the data. The process at shorter times shows a close similarity the methyl group rotation found for polymeric systems. The process at longer times was assigned to glassy dynamics. The obtained data are compared in detail in their temperature dependence to the dielectric results.
Dynamics of polymer melt in nanoconfinement

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Keywords: neutron, polymer dynamics, neutron spin echo

Polymer dynamics is very rich and shows different phenomena depending on the temperature and length scale of observation. Close and above the glass transition temperature \(T_g\), the dynamics is dominated by the \(\alpha\)-relaxation at inter-molecular level. Increasing the temperature, chain mobility is enhanced and, at length scales larger than those corresponding to the statistical segment, the chain dynamics can be well described by the well-established model.

Confinement of polymers by a solid surface on the nanometer scale leads to variety of the structural and dynamical effects that are crucial for nanotechnology. In particular, functional polymeric nanomaterials typically consist of nano-sized particles, which are homogenously dispersed in a polymeric matrix (nanocomposites). Alternatively, the polymer chains are confined in porous media, e.g. in long cylindrical nanopores (nanoconfinement). In both cases, presence of a large amount of solid surface reduces the number of possible conformations of a polymer chain and influences the dynamics, in particular for those macromolecules, which are close to the surface [1].

In a neutron study we have shown that the attractive polymer-surface interaction influences polymer dynamics. In particular, adsorption of polymer to the Alumina pore surface led to the formation of a surface layer with the dynamics slowed down as compared to the dynamics of pure polymer [2]; anchoring of polymer segments on the Alumina surface creates an interphase between the polymer in close vicinity to the solid surface and pure polymer [3]. In addition, at strong confinement conditions the dilution of the entanglement network is observed [4]. Here we also focus on influence of the confinement on the polymer dynamics in absence of specific adsorption of the polymer segment on the Alumina surface, i.e. repulsive or neutral polymer-surface interaction will be considered.

[1] Polymer Interfaces and Interphases, Soft Materials: Special Issue. 12 (2014); DOI: 10.1080/1539445X.2014.964803
Lipid Bilayer Dynamics Confined by Single-Sided Hard Wall

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Keywords: lipid bilayers, confinement, grazing incidence scattering, neutron spin echo spectroscopy

Lipid bilayers serve as model membranes that appear manifold in living organisms at various places. The observation of membrane-stack relaxations at solid-liquid interfaces is highly interesting because such stacks support the low friction movements of cartilage in joints. The results from grazing incidence neutron spin echo spectroscopy measurements on such membrane stacks explain how point-like excitations are dissipated over wider distances and so local membrane destruction is minimized.

We studied soyPC lipid bilayers that were swollen by heavy water in the vicinity to a solid interface. The newly developed resonator is a neutron wave guide at a large silicon slab, consisting of three double layers. Through constructive interference at small incidence angles, well below the critical angle of total reflection, the neutron intensity is dramatically enhanced and so the evanescent wave highlights the near-surface dynamics of the membrane stack. Using a neutron spin echo spectrometer, the nanosecond dynamics are perfectly resolved. The asymmetric incidence and exit angles serve for a small in-plane scattering vector that highlights long-wavelength membrane modes. Astonishingly, we found propagating wave modes along the surface besides the otherwise dominating over-damped membrane undulations. This viscoelastic twofold behavior on the nanosecond time scale is for the first time observed in neutron spin echo spectroscopy for a Soft Matter system: Oscillating relaxation curves are obtained.

Apart from the bare lipid system we were interested on the influence of the additive ibuprofen. While high ibuprofen contents destroy the lamellar stack structure (that explains stomach bleeding for long application times), the dynamics show fewer wave modes at intermediate concentrations and again more wave modes at highest concentrations.
Posters will be up during the entire conference. Software will not only be presented on posters, but also live on computers.

For the list of posters and abstracts, please refer to page 109 and following pages in the end of this document.
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Topological Aspects of Magnetic Order

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Keywords: neutron, magnetic order, topology

Many decades of intense research based on the notions of symmetry breaking and generalized rigidities have resulted in a remarkably comprehensive account of complex forms of magnetic order in condensed matter systems. In recent years a new facet of magnetism research receives increasing attention that concerns the topological character of magnetically ordered systems, notably those properties that remain unchanged under elastic deformations. Important examples include skyrmions, vortices and monopoles in chiral or frustrated magnets. These topological aspects of magnetic order are not only appealing from an esthetical and conceptual point of view, but offer strikingly simple explanations for materials properties that may seem to be surprising and hideously complicated at first sight.
Small-angle neutron scattering (SANS) is a very powerful technique for the investigation of magnetic materials, since it provides information from within the bulk of magnetic media and on a length scale between a few nanometers and a few hundred of nanometers ($\sim$ 1-300 nm). In this talk, we summarize recent theoretical and experimental work in the field of magnetic SANS of bulk ferromagnets.\(^1\) The response of the magnetization to spatially inhomogeneous magnetic anisotropy and magnetostatic stray fields is computed using micromagnetic theory, and the ensuing spin-misalignment SANS is deduced. This approach — originally pioneered by Kronmüller, Seeger, and Wilkens (Ref. 2) — goes beyond the traditional description of SANS in terms of particle form and structure factors. Analysis of experimental magnetic-field-dependent SANS data corroborates the usefulness of the approach, which provides important quantitative information on the magnetic-interaction parameters such as the exchange-stiffness constant, the Dzyaloshinski-Moriya interaction, the mean magnetic anisotropy field, or the mean magnetostatic field due to jumps $\Delta M$ of the magnetization at internal interfaces. Besides the value of the applied magnetic field, it turns out to be the ratio of the magnetic anisotropy field $H_p$ to $\Delta M$, which determines the properties of the magnetic SANS cross section of bulk ferromagnets; specifically, the angular anisotropy on a two-dimensional detector (see figure below), the asymptotic power-law exponent, and the characteristic decay length of spin-misalignment correlations. Unpolarized and polarized neutron data on hard and soft magnetic nanocomposites will be discussed.

(a)-(d) Contour plots of the normalized magnetic SANS cross section at applied magnetic fields as indicated ($k_0 \perp H_0$); $H_0$ is horizontal in the detector plane. The change in angular anisotropy with increasing field that becomes visible in (a)-(d) reflects the competition between anisotropy-related and magnetostatic correlations. (e)-(h) Corresponding two-dimensional correlation functions $C(y,z)$ (for further details see Ref. [1]).

References:


Magnetization Distribution and Shape Induced Aggregation Behavior in Magnetic Nanoparticles

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Keywords: magnetic nanoparticles, surface spin disorder, self-organization

Magnetic nanoparticles reveal unique magnetic properties which make them relevant for data storage, electronic and mechanical engineering, and biomedical applications\textsuperscript{1,2}. With regard to these applications, the main aspects of fundamental interest include magnetic anisotropy and the related magnetization distribution in individual nanoparticles as well interparticle interactions leading to aggregation or even ordered assemblies of nanoparticles.

Shape anisotropy has a substantial influence on the nanoparticle magnetization and magnetization relaxation as well as interparticle interactions. In this contribution, we will give an overview of our studies of spherical and cubic iron oxide nanoparticles aiming at both \textit{intra}particle magnetization and \textit{inter}particle correlations. Particular emphasis is on the impact of particle shape and size on the spatial magnetization distribution and surface spin disorder\textsuperscript{3,4} as well as the aggregation behavior of cubic particles in concentrated dispersion. Geometric orientation and agglomeration of the particles will be discussed as suggested by field-dependent SANS data.

References:
DOII:10.1088/1742-6596/711/1/012002
Session 6: Magnetism/Superconductors II

Thursday, Sept. 22, 2016, 11:00 - 12:30. Session Chair: Thomas Brückel
Location: Frederik-Paulsen-Hörsaal

11:00 - 11:15: Switching from Sign-reversed to Sign-preserved Cooper-pairing Symmetry in Sulfur-doped Iron Selenide Superconductors (ID 205) Jitae Park, Qisi Wnag, Yu Feng, Yao Shen, Bingying Pan, Jeff Lynn, Alexander Ivanov, Songxue Chi, M Matsuda, Huibo Cao, Robert Birgeneau, Dmitri Efremov, Jun Zhao

11:15 - 11:30: Magnetic Phase Diagram of CeCu$_2$Ge$_2$ up to 15 T - New Input to Understand Neutron Scattering Results (ID 239) Astrid Schneidewind, Philipp Geselbracht, Mathias Doerr, Martin Rotter, Michael Loewenhaupt, Gernot Scherer, Zhi Ren, Prokes Karel

11:30 - 11:45: Neutron Diffraction Studies of Fe Substituted Spinel Cobalt Chromite (ID 125) Ram Kumar, R. Padam, S. Rayaprol, V. Siruguri, D. Pal

11:45 - 12:00: Non-collinear Magnetic Structure in Cobalt Chromite Nanoparticles (ID 137) Dominika Zákutná, Jan Vlček, Přemysl Fitl, Stefan Roitsch, Kirill Nemkovski, Yixi Su, Daniel Nižňanský, Sabrina Disch

12:00 - 12:15: Magnetic Properties and Spin Structure of MnO and FePt@MnO Nanoparticles (ID 118) Xiao Sun, Alice Klapper, Yixi Su, Kirill Nemkovski, Andrew Wildes, Heiko Bauer, Oskar Köhler, Anna Schilmann, Wolfgang Tremel, Oleg Petracic, Thomas Brückel

12:15 - 12:30: Chirality Induced Exchange Bias Effect in DyCo/FeNi Bilayers (ID 207) Dieter Lott, Kai Chen, Valeria Lauter
Switching from sign-reversed to sign-preserved Cooper-pairing symmetry in sulfur-doped iron selenide superconductors

Qisi Wang, Jitae Park, Y. Feng, Y. Shen, Y. Hao, B. Pan, J. W. Lynn, A. Ivanov, S. Chi, M. Matsuda, R. J. Birgeneau, D. V. Efremov, Jun Zhao

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Keywords: iron-based superconductors, spin resonance mode, inelastic neutron scattering

An essential step toward elucidating the mechanism of superconductivity is to determine the sign/phase of superconducting order parameter, as it is closely related to the pairing interaction. In conventional superconductors, the electron-phonon interaction induces attraction between electrons near the Fermi energy and results in a sign-preserved s-wave pairing. For high-temperature superconductors, including cuprates and iron-based superconductors, prevalent weak coupling theories suggest that the electron pairing is mediated by spin fluctuations which lead to repulsive interactions, and therefore that a sign-reversed pairing with an s± or d-wave symmetry is favored. Here, by using magnetic neutron scattering, a phase sensitive probe of superconducting gap, we report the observation of a transition from the sign-reversed to sign-preserved Cooper-pairing symmetry with insignificant changes in $T_c$ in the S-doped iron selenide superconductors $K_xFe_{2-y}(Se_{1-z}S_z)2$. We show that a rather sharp magnetic resonant mode well below the superconducting gap ($2\Delta$) in the undoped sample ($z = 0$) is replaced by a broad hump structure above $2\Delta$ under 50% S doping. These results cannot be readily explained by simple spin fluctuation-exchange pairing theories and, therefore, multiple pairing channels are required to describe superconductivity in this system. Our findings may also yield a simple explanation for the sometimes contradictory data on the sign of the superconducting order parameter in iron-based materials [1].

References:
Magnetic phase diagram of CeCu$_2$Ge$_2$ up to 15 T - new input to understand neutron scattering results

P. Geselbracht$^1$, M. Doerr$^2$, S. Granovsky$^2$, M. Rotter$^2$, M. Loewenhaupt$^2$, G. W. Scheerer$^3$, Z. Ren$^3$, G. Lampertot$^3$, K. Prokes$^5$, Astrid Schneidewind

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Keywords: correlated electron systems, phase diagram, phase transition, magnetic order

The magnetic ($H,T$)-phase diagrams of CeCu$_2$Ge$_2$ and CeCu$_2$Si$_2$ have similar features, although only the latter one becomes superconducting at ambient pressure. In tetragonal CeCu$_2$Ge$_2$ at least three magnetic phases AF1, AF2 and a yet unknown phase IM exist below $T_N = 4.2$ K and in fields up to 35 T. Furthermore, the nature of the phase transition from AF1 to AF2 is discussed controversially in the literature. We present results for fields up to 15 T parallel to the [1-10] direction of thermodynamic (magnetization, magnetostriction), electrical transport and elastic neutron diffraction measurements.

At temperatures below 1 K a first order transition from AF1 to AF2 at around 8 T was found characterized by a change of the propagation vector from $q_1 = (0.285, 0.285, 0.538)$ to $q_2 = (0.310, 0.310, 0.543)$, followed by another slight shift at 10 T to $q_{2\text{mod}} = (0.317, 0.317, 0.543)$. Above 12.5 T the magnetic satellites of type (HHL) have vanished.

To interpret the results of macroscopic measurements and existing neutron scattering data a mean-field simulation by the McPhase program was realized, with following results:

At low temperatures and in zero field a propagation of $q_{1\pm} = (0.278 \pm 0.278, 0.556)$ is stabilized and a double-$q$ structure is obtained. The propagation vector and the predicted shifts with field and temperature have been calculated. The double-$q$ type of magnetic order is stable in magnetic field up to ~9 Tesla. Above this field single $q$ structures are stabilized, the propagation varies with field. The disappearance of the magnetic satellites in the experimental data may be interpreted in a natural way by the transition from the double-$q$ to the single-$q$ magnetic structure. Note that in the experiment with (HHL) scattering plane only the satellites of $q_1$ may be observed and therefore no peaks have been found in the single-$q$ phase region. The theoretical analysis predicts an AF2 double-$q$ structure with $q_{2\pm} = (0.286, \pm 0.286, 0.556)$ at higher fields and temperature. This increase in $H$ is in agreement with the experimental observation.

These calculations also reveal the principal shape of magnetization and susceptibility curves published earlier. Finally, the calculated ($H,T$)-phase diagram agrees satisfactorily to that deduced from experiments. This consistence of experimental results and theoretical model is important for the understanding of magnetism in CeCu$_2$Ge$_2$.
Neutron Diffraction Studies of Fe Substituted Spinel Cobalt Chromite

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Keywords: neutron diffraction, magnetic oxides, ferrimagnetics.

Spinel cobalt chromite, CoCr$_2$O$_4$ is a conical spiral magnet that has attracted considerable attention due to magnetically and thermally reversible ferroelectric polarization stemming from the gigantic coupling between spin and charge dependent properties. In order to see the influence of substitution, we have chosen Fe to substitute at Cr site. We report here the study of structural and magnetic properties of the multiferroic compound CoCr$_2$O$_4$ on partial substitution (5% and 7.5%) of Cr by Fe. By studying the magnetic properties and magnetic structure by performing neutron diffraction experiment of the Fe substituted samples one can see how magnetic transitions are getting affected, while crystal structure remains unchanged.

Six panels of Fig.1 depict the comparison of the temperature dependent spontaneous magnetization obtained from the refinement of the magnetic phase of the Neutron Diffraction (ND) patterns of 5% and 7.5% Fe samples with that of the bulk magnetization measured experimentally under the applied fields of 0.01T and 5T. We can see that, similar to the bulk magnetization, the moments obtained from the ND data also exhibit near zero values at the compensation points $T_{\text{comp}} \approx 43.8\,\text{K}$ and $\approx 67.8\,\text{K}$ for 5% and 7.5% Fe, respectively. And remarkably, it can be observed that the behavior of the spontaneous magnetization across the compensation point is very similar to the bulk magnetization under 5T field. One more point that is clear from these combined plots is that magnetization obtained from both measurements is of the same order for the compounds. From this, it is understood that there are orientations of the moments corresponding to different sub-lattices, across the compensation point. All these features are not noticed in case of the parent CoCr$_2$O$_4$ compound.

References:
Non-collinear Magnetic Structure in Cobalt Chromite Nanoparticles

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Keywords: xyz polarization analysis, spiral structure, propagation vector, magnetic nanoparticles

Cobalt chromite has attracted great scientific interest due to its effective magneto-electric coupling known as magnetoelectric effect 1 . The appearance of dielectric polarization in cobalt chromite is associated with a non-collinear magnetic structure. Up to date, the presence of spiral magnetic ordering has been observed in single crystalline 1-2 and polycrystalline 3-4 cobalt chromite. In this contribution, we will present the observation of spiral magnetic ordering in cobalt chromite nanoparticles with respect to their particle size.

We have determined the magnetic properties of nanosized cobalt chromite on both the macroscopic and microscopic level by a combination of magnetization measurements and neutron diffraction with xyz polarization analysis. The prepared cobalt chromite nanoparticles with mean particle diameter of 26.9(1) nm ($\sigma_{\log} = 23\%$), exhibit ferrimagnetic ordering below 92 K. In contrast to the bulk material, with a transition from paramagnetic to long-range ferrimagnetic ordering, the synthesized cobalt chromite nanoparticles reveal magnetic phase transitions from paramagnetic through superparamagnetic (SPM) to ferrimagnetic ordering. The macroscopic magnetization measurements reveal also a phase transition around 25 K, similar to the transition to spiral magnetic ordering observed in the bulk material 1-4 . The clear presence of spiral magnetic ordering in our nanoparticles was confirmed by neutron diffraction with xyz polarization reflections below the transition to the non-collinear structure, $T_s < 25$ K. We will present our evaluation of the non-collinear magnetic structure according to the spiral model proposed by Lyons, Kaplan, Dwight, Menyuk 4 and Hastings, Corliss 5 . The detailed spin structure including the temperature dependence of the spiral propagation vector as well as the impact of a significantly smaller particle size (3.1(2) nm diameter) will be discussed in detail.

References:

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Magnetic Properties and Spin Structure of MnO and FePt@MnO Nanoparticles

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Keywords: neutron, magnetism, nanoparticles

The spin structure of antiferromagnetic (AF) MnO nanoparticles (NPs) has been investigated for various sizes (5-20nm) using both magnetometry and polarized neutron scattering. Zero field cooled (ZFC) magnetization curves show a peculiar peak at low temperatures (ca. 25K) instead at the T_N of 118 K (Fig. 1(c)). However, the AF order parameter of MnO shows the expected behavior from polarized neutron scattering studies. To resolve this apparent contradiction further magnetometry studies using ZFC/Field Cooled (FC) curves at various fields, memory effect and susceptibility measurements were employed. The results can be explained by a superposition of superparamagnetic-like thermal fluctuations of the AF Néel vector inside an AF core and a strong magnetic coupling to a Mn_2O_3 or Mn_3O_4 shell¹.

We have also studied the magnetic influence of ferromagnetic (FM) FePt particles in direct contact to MnO NPs. Neutron scattering results on the FePt@MnO dimer NPs show a comparable behavior to the MnO NPs. I.e. the AF order parameter vanishes at a temperature between 100 K and 140 K. However, an additional exchange bias is observed, which is explained by the exchange interaction of the MnO NP shell to the FePt subunit.

These results provide a closer insight into the spin structure and interactions of more complex NP configurations (i.e. dimers), which can possibly be used for novel nanomaterials with tailored physical properties or energy efficient nanomagnetic devices.

Fig.1: Separated neutron scattering contributions of (a) 12nm MnO NPs and (b) FePt@MnO NPs with an average size of 6nm@12nm from polarized neutron scattering. (c) ZFC/FC curves of 12nm MnO NPs.

References:

Chirality induced exchange bias effect in DyCo/FeNi bilayers

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Keywords: polarized neutron reflectometry, polarization analysis, chirality, exchange bias

Alloys of rare-earth elements and 3d transition metals became recently again in the focus of attention due to their rich variety of magnetic effects owed to the different anisotropies of both material classes [1-4]. In this work, a dual in-plane exchange bias effect (HEB=±39mTesla) were found in the orthogonal coupled thin Dy20Co80/NiFe bilayer occurring at room temperature without the need of any field cooling procedure. Particularly interesting, the direction of the exchange bias effect can be switched by changing solely the direction of the perpendicular magnetic fields in relative moderate fields of about 200mT (see Fig. 1).

The in-plane exchange bias keeps stable even at external in-plane magnetic fields of more than 6T. The underlying mechanism behind the extraordinary effect was investigated with magnetic optical Kerr effect (MOKE), X-ray magnetic circular dichroism (XMCD) and polarized neutron reflectometry measurements (PNR). Particularly the use of polarized neutrons enabling one to get access to the chemical and magnetic depth profile was essential to identify chirality as the crucial mechanism behind the intriguing isothermal exchange bias effect. The application of polarization analysis allowed us to analyze the non-collinear magnetic structure during the switching behavior in detail. The exchange bias in NiFe can be attributed to the interface exchange coupling to the DyCo layer, while the exchange bias in DyCo layer is due to the formation of chirality in its spin structure formed during the deposition on NiFe. Latter indicates a Dzyaloshinskii-Moriya like term in the interacting energy originating from the broken inversion symmetry at the interface between both magnetic layers. Such chirality based exchange bias systems may be of crucial importance for the development of future applications in the field of magnetic sensors.

M4_2: Minisymposium “Neutron spectroscopy for dynamics in confinement”, part 2

Thursday, Sept. 22, 2016, 11:00 - 12:30. Session Chair: Michael Marek Koza
Location: Hörsaal D

11:00 - 11:20: **Diffusion of Hydrogen in Nanoporous and Exfoliated Carbons** (ID 267) *Peter Fouquet*


11:40 - 12:00: **Water Dynamics in Clays** (ID 216) *Fanni Juranyi, Thomas Gimmi, Martina Bestel, Sergey Churakov, Luc Van Loon*

12:00 - 12:20: **Neutron Scattering Advances Dental Cement Research** (ID 185) *Marcella Berg, Ana Benetti, Heloisa Bordallo*
Diffusion of hydrogen in nanoporous and exfoliated carbons

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Keywords: neutron, porous materials, QENS, carbon, hydrogen

The potential use of porous carbons and carbon nano-structures for hydrogen storage has motivated a large number of investigations in recent years, but only few studies have used neutrons to look at the microscopic dynamics of H₂.

In our recent studies [1], we have focused on the hydrogen adsorption and transport properties of carbon gels prepared from resorcinol-formaldehyde resins and on exfoliated graphite. To compare the effect of the surface chemistry and pore characteristics, samples were prepared with different drying methods and post carbonization treatments. The carbon gels were characterized by low temperature N₂ adsorption/desorption, CO₂ adsorption and X-ray photoelectron spectroscopy (XPS). H₂ uptake measurements were performed at different temperatures. The mobility of the adsorbed H₂ on the surface was studied by backscattering (BS) and neutron spin-echo (NSE) spectroscopy.

Our experiments show that the combination of TOF and NSE spectroscopy is a very powerful tool to study the diffusivity of adsorbed H₂, even at low relative coverage. The results reveal that the surface chemistry of the carbon has huge impact on the surface diffusion properties of the hydrogen. On the other hand desorption of the hydrogen can be shifted towards significantly higher temperatures by changing the pore characteristics of the carbon.

References:
Quasielastic neutron scattering studies on propane in nanoporous media

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Keywords: quasielastic neutron scattering, dynamics in confinement, propane dynamics

In natural environments, hydrocarbons are usually found confined in porous media. Confinement, temperature, pressure and presence of other species in the nanopores, influence the dynamics of confined hydrocarbons. We report quasielastic neutron scattering studies of these effects on propane dynamics. The dynamical properties of the system were studied as a function of temperature and pressure as well as the presence of CO₂ and D₂O. Because of the high incoherent scattering cross-section of hydrogen, major contribution of the signal arises from propane. The effects of pressure changes are found to be more pronounced than the effects of temperature. At low pressures of propane, many propane molecules are adsorbed onto the pore surfaces and are thus immobile. As the pressure of propane loading is increased, more molecules become available to diffuse inside the pores. At low pressure these mobile propane molecules undergo continuous diffusion, while at higher pressures, the propane molecules exhibit distinct jumps. In media with pore size of approximately 20 nm, presence of CO₂ enhances the jump rate of propane molecules, thereby increasing the diffusion coefficient. On the other hand, in media with poresize of about 1.5 nm, the presence of D₂O slows down propane diffusion. This study aims to aid in understanding the complex processes involved in hydrocarbon migration in porous quartz-rich rocks and enhanced hydrocarbon recovery.
Water Dynamics in Clays

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Keywords: swelling, water distribution, water diffusion, observation time

Context: Clays are layered materials with complex hierarchical pore structure. Beside their importance in applications (waste management, filtration, food industry ...) they are often used as host materials to understand confinement effects. Water diffusion at different time and length scales plays a crucial role in this context.

Aim: For several applications, diffusion on the macroscopic scale is the property of interest. It depends on local physico-chemical interactions as well as on the pore structure of the material. Therefore we aim to understand the diffusion process in these complex hierarchical materials at different scales and link them together. The situation is most challenging in case of swelling clays, where water can be located in different type of pores, and have different properties. Contrary to the non-swelling clays, here the measured activation energy differs on the macroscopic and the atomic scales\textsuperscript{1}.

Results: According to simulations the interlayer distance and average particle size are the relevant length scales for the measured diffusion process\textsuperscript{2}. Quasielastic neutron scattering was used to measure water diffusion at the atomic scale in bi-hydrated Na-montmorillonite as a function of observation time. The transition from a local 3D into a 2D diffusion within the interlayer could be observed for the first time\textsuperscript{2}. When the water to clay ratio is higher, free water in the interparticle pores appears. Elastic incoherent neutron scattering was found to be a useful method to measure the ratio of interlayer and interparticle water. In this case the QENS signal is additive. Opposite to free water, water confined in the interlayer does not crystallize, but was found to undergo a glass transition. Importantly, the measured phonon density of states differs from that of the amorphous ice because of the 2D confinement.

References:


The development of dental materials with improved properties and increased longevity can save costs and minimize discomfort for patients. Due to their good biocompatibility, glass ionomer cements (GIC) are an acceptable alternative material for preventive dentistry. However, these cements have limited mechanical strength to survive sufficiently long in the challenging oral environment. As dental cements are complex systems, where important motions related to the different states of the hydrogen atoms evolve in a confined porous structure, a better understanding of their hydration process and liquid mobility is important for further development of these functional materials.

In this regard, neutron spectroscopy has been used to assess the hydrogen dynamics within different GIC cements. The observed differences between the investigated materials were expected due to their distinct formulation and powder to liquid ratio. Nonetheless, the dissimilar mobility observed in the GIC cements helps to further the understanding of the relationship between microstructure, liquid dynamics in confinement and strength. Our results bring invaluable insights into the material’s durability, also demonstrating the need for, and opening the possibility for further research in these dental cements.

References:

P-2 (ID 193): Neutron Optics from HZB. Thomas Krist, Jennifer Schulz, Jan Hoffmann


P-4 (ID 134): KOMPASS - a Triple-Axis Spectrometer Optimized for Polarization Analysis. Alexander Grünwald, Jonas Stein, Georg Waldherr, Stefan Giemsa, Peter Böni, Markus Braden

P-5 (ID 130): Recent Developments at DNS, Diffuse Neutron Scattering Spectrometer with Polarization Analysis at MLZ. Yixi Su, Kirill Nemkovskiy, Sultan Demirdis, Werner Schweika, Alexander Ioffe, Thomas Brückel

P-6 (ID 143): Further Development of an Experimental Setup for Investigation on Magnetic Thin Layers by in-situ Neutron Reflectometry. Jingfan Ye, Wolfgang Kreuzpaintner, Birgit Wiedemann, Sina Mayr, Andreas Schmehl, Thomas Mairoser, Alexander Herrnberger, Jean-François Moulin, Jochen Stahn, Panos Korelis, Martin Haese, Matthias Pomm, Amitesh Paul, Peter Böni, Jochen Mannhart

P-7 (ID 234): Searching for an Optimal Binning Strategy for Reflectometry Data. Jean-François Moulin, Sebastian Busch, Martin Haese


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P-11 (ID 289): Polarized Neutron Reflectometer NERO for Reactor PIK. 

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Peter Konik, Sergey Grigoriev, Evgeny Moskvin


P-17 (ID 189): Novel Type of Polarisation Analysis With the Multianalyser at PUMA. Steffen Schwesig, Oleg Sobolev, Götz Eckold

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P-22 (ID 286): **Neutron Stress-Diffractometer ARES for PIK Reactor.** Aleksei E. Sokolov

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P-37 (ID 119): Chopper System of the Beamline for European Materials Engineering Research (BEER) at ESS. Mustapha Rouijaa, Jochen Fenske, Peter Staron, Gregor Nowak, Martin Mueller, Heinz-Günter Brokmeier, Jan Šaroun, Premysl Beran, Petr Lukas, Petr Šittner

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P-78 (ID 186): Change-over from Linear to Branched Growth During the Nucleation Phase of Lysozyme Crystallization. Raimund J. Heigl, Jörg Stellbrink, Aurel Radulescu, Ralf Schweins, Tobias Erich Schrader, Dieter Richter

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P-86 (ID 182): The Method of Soil Moisture Sensing by Cosmic Ray Neutrons. Markus Köhli, Martin Schrön, Steffen Zacharias, Peter Dietrich, Ulrich Schmidt

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P-88 (ID 209): Porphyrin on Semiconductor Nanoparticles as Seen by Combined X-ray and Neutron Small Angle Scattering. Torben Schindler, Jano Heppt, Tilo Schmutzler, Tobias Unruh


P-90 (ID 261): Encoding the Shape of Nano-Assemblies: A SANS-Thermodynamics Study as Key to a Fundamental Understanding of Structure Directing Effects. Franziska Gröhn, Mariani Giacomo, Schweins Ralf
P-91 (ID 225): **Self assembly and structural properties of magnetic nanoparticles on silicon surfaces.** *Katharina Theis-Bröhl, Philipp Gutfreund, Max Wolff, Apurve Saini, Alexei Vorobiev, Erik Brok, Kathryn Krycka, Joseph A. Dura, Julie A. Borchers, Erika C. Vreeland, Dale Huber*

P-92 (ID 140): **The Structure of MAPbI₃: a Neutron and Synchrotron X-ray Diffraction Study.** *Alexandra Franz, Daniel M. Többens, Susan Schorr*

P-93 (ID 128): **Lattice Thermal Expansion of Bi₂Fe₄O₉, Bi₂Fe₂Mn₂O₁₀ and Bi₂Mn₄O₁₀.** *M. Mangir Murshed, C.M.N Kumar, Ashfia Huq, Thorsten M. Gesing*

P-94 (ID 183): **Overview of Single crystal Diffraction Studies with HEiDi.** *Martin Meven, Andrew Sazonov, Vladimir Hutanu, Georg Roth*

P-95 (ID 250): **Neutrons on the Web - the KFN Website „sni-portal.de/kfn“.* *Karin Griewatsch*
Advanced neutron optics on the new IN12

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Keywords: neutron, instrumentation, optics, polarization, three-axis

IN12, a three-axis spectrometer for cold neutrons, is operated as a CRG-instrument from the Jülich Centre for Neutron Science (JCNS) at the Institute Laue Langevin in Grenoble. In the framework of the Millenium Program of the ILL IN12 has been relocated to a new position at the end of a new guide. Along with this relocation the whole primary spectrometer has been upgraded with new state-of-the-art components [1].

The main improvements concern a new optimized focusing neutron guide together with a new double focusing monochromator. Compared to the old IN12 this combination of a modern neutron guide and the focusing Bragg optics has yielded up to a factor of 10 in flux on the sample position. We can now offer a peak flux of about $10^8$ n/sec/cm² around $k_i = 2 \, \text{Å}^{-1}$. In addition an extended wavelength range far into the warmish region (max. $k_i = 5.1 \, \text{Å}^{-1}$) is now available.

Due to the large wavelength range the higher order reflections from the monochromator would of course be much more prominent. Therefore a neutron velocity selector is placed upstream in the neutron guide that prevents all these contaminations and guarantees a clean beam at the sample.

For the use of polarized neutrons a new transmission polarizer (cavity) has been installed in the neutron guide, mounted on a guide changer together with a standard guide element. This guarantees high intensities and an easy change from non-polarized to polarized mode.

In this presentation we will show details of the design and optimization of the various neutron optical components. This includes the results of various model calculations that had been performed to match a curved focusing guide end with a double focusing monochromator, in order to combine high flux, good energy resolution and a smooth homogenous beam profile at the sample. Further, also the results for the design of the polarizing cavity will be presented.

The new IN12 is back in normal user operation and we will show neutron measurements and results from user experiments that demonstrate the capabilities of this powerful three-axis spectrometer.

Neutron Optics from HZB

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Keywords: neutron, instrumentation, polarization, focusing

At HZB we have developed several neutron optical devices, mainly solid state elements where the neutrons are transported in thin silicon wafers with coated walls.

Results are shown of solid state polarizing benders, solid state collimators and a solid state radial bender for the polarization analysis of neutrons over an angular range of 3.8 degrees.

Another device consists of a solid state polarizing bender without absorbing layers used together with a collimator, which allows polarizing or analyzing neutrons without deflecting them from their original direction.

Two-dimensional polarization analysers for an angular range of 5 degrees in both directions are presented. Another device consists of a solid state spin splitter and a collimator, which allows polarizing or analyzing neutrons without deflecting them.

In all these polarizing devices polarisations of 95% were realised.

In the last year several polarizing S-benders were built with very high polarizations, e. g. one with a cross section of 30 mm x 100 mm was tested at a wavelength of 4.4 Å, showing a polarization above 98% and a transmission above 65%.

A focusing solid state lens was made from Si wafers coated with m=2 Ni-Ti supermirrors which can transport the full divergence of a neutron guide with the same coating. Here a focus with a FWHM of 2.4 mm was reached and an intensity increase of 5.6 compared to the intensity there without the lens.

Refractive focusing of neutrons can be realized by prisms. We used Si prism systems to analyse the energy of a collimated white neutron beam and to refocus a 0.2 mm wide slit onto a detector where a FWHM of 0.35 mm was reached and the maximum intensity there was 9.7 times higher than the direct beam.

References:
FALCON - A Laue Diffractometer for ambient and non-ambient Neutron Structural Analysis

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Keywords: Laue diffraction, single crystal neutron diffraction

End of 2015 the FALCON Laue diffractometer at the Berlin neutron source BERII started. It was developed in collaboration with the ILL, Grenoble. The diffractometer is designed for fast neutron scattering data acquisition of single crystals and makes use of a white (“pink”) neutron beam with wavelength band of 0.8-3.2 Å. Pattern acquisition is performed by means of a backscattering and a transmission detector consisting of four iCDD cameras each. The detectors cover a read active area of 400x400 mm². Acquisition time depending on sample is about half up to three minutes. The sample is mounted on an Irelec cradle allowing sample positioning and orienting. A chamber for sample cooling is in progress and will open up the possibility for Laue data acquisition down to temperatures of about 60K. The instrument set-up as well as some worked examples will be shown in detail.

Fig. 1: FALCON neutron Laue diffractometer
Fig. 2: Laue image of ruby Al₂O₃

References:
KOMPASS – a Triple-Axis Spectrometer Optimized for Polarization Analysis

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Keywords: neutron, instrumentation, structure, dynamics, polarization analysis

Abstract: The new triple-axis spectrometer at the FRM II – KOMPASS [1] – is designed to work exclusively with polarized neutrons. It is optimized for zero-field spherical neutron polarization analysis for measuring all elements of the polarization matrix. In combination with its end position at the cold neutron guide NL-1, the doubly focusing monochromator and analyzer mechanics equipped with highly oriented pyrolytic graphite, the instrument will be very well suited to study various types of weak magnetic order and excitations, including the possibility to conduct time-resolved and stroboscopic measurements, e.g. studying the domain distribution when switching an electric field in multiferroics.

In addition to the global instrument concept, we will present the variable guide design and the state-of-the-art polarization concept, using multiple cavities, which will provide a highly polarized beam [2, 3]. The unique feature of KOMPASS is a permanently installed triple cavity in the incoming beam. For the polarization analysis of the scattered neutrons, the secondary spectrometer can be equipped with a combination of a cavity of trapezoidal geometry and a solid-state-collimator to tune the performance in terms of polarization and transmission. A couple of pictures will document the mounting and commissioning of the instrument.

References:
Recent developments at DNS, diffuse neutron scattering spectrometer with polarization analysis at MLZ

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Keywords: neutron polarization analysis, diffuse scattering, instrumentation

DNS is a versatile diffuse scattering instrument with polarization analysis operated by JCNS at the Heinz Maier-Leibnitz Zentrum (MLZ). A compact design, a large double-focusing PG monochromator and a highly efficient supermirror-based polarizer provide a polarized neutron flux of about $10^7$ n/cm$^2$s. DNS is used for the studies of highly frustrated spin systems, strongly correlated electrons, emergent functional materials and soft condensed matter.

Here we present the recent developments and scientific highlights at DNS as well as the ongoing instrument upgrades. In the frame of this project, the neutron guide with m=1.2 has been replaced by the new one with m=2. Respectively, the flux for the short-wavelength range has been considerably enhanced. The available short wavelength range has been extended from 2.4 Å to 1.5 Å, with an accessible Q-range up to 7.8 Å$^{-1}$ instead of 4.8 Å$^{-1}$. In order to deal with the increased neutron flux, the monochromator shielding has been replaced and improved. In addition, a neutron velocity selector has been installed and successfully commissioned. It allows us to suppress the high-order contamination of the monochromatized beam. Alternatively, the velocity selector can be used for selecting a shorter wavelength by the PG004 reflection with better resolution and without moving the secondary spectrometer. An important step toward a user-friendly instrument has been achieved by switching to the new generation instrument control software TANGO and NICOS. The new option for the data reduction and visualization in diffraction mode has been developed and implemented based on the Mantid project.
Further Development of an Experimental Setup for Investigation on Magnetic Thin Layers by in-situ Neutron Reflectometry

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Keywords: in-situ, neutron reflectometry, magnetism, thin films, sputtering

Thin magnetic layers are used in many magneto-electronic devices. A thorough understanding of their texture and the coupling between them is, therefore, essential to improve functionality. In order to avoid the incorporation of impurities and surface contamination, these layers have to be grown under ultra-high-vacuum (UHV) conditions. An excellent method to analyze their structural and magnetic properties is polarized neutron reflectometry (PNR). Up to now, the sample growth and analysis have been performed in separate steps. The grown samples had to be removed from the vacuum after the growth process and put into the neutron beam line for analysis. This procedure is time consuming and leads to the sample being exposed to the environment which could alter their properties.

In this contribution, a compact in-situ vacuum sputtering chamber with integrated DC and RF sputtering equipment for performing in-situ neutron reflectometry experiments will be presented. It can be easily transported and installed at neutron beam lines and combines both, deposition and measurement, into one single setup. This allows magnetic properties of thin films as a function of their thickness and composition to be investigated, both, quickly and on one single sample, at different evolutionary deposition steps.

Despite the compactness of the device, no compromises regarding conditions for growth and measurement were made: A coil allows field dependent measurements and a cryostat with integrated heater can set the sample temperature in a range from 10K to 1000K. We constructed our device to be fully compatible to the REFSANS (FRM II) and the AMOR (PSI) ToF neutron reflectometers. At PSI, we can perform PNR measurements using the state-of-the-art SELENE neutron optical setup which reduces measurement times from hours to several minutes.

The results of very promising measurements on Fe/Pd, Fe/Cr and Fe/Cu layers are shown in contributions by Sina Mayr and Wolfgang Kreuzpaintner.
Searching for an Optimal Binning Strategy for Reflectometry Data

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Keywords: neutron, data analysis, reflectometry

In all fields of science where spectra need to be fitted one has to choose the optimal data binning strategy in order to extract the maximum amount of information from the experiments. Obvious criteria are related to the minimal intensity in each data bin and the actual instrumental resolution. Nevertheless naming these criteria is not enough and it is quite usual that some ad-hoc methods are used to define the binning. Ultimately, the choices are often based on experience, somewhat arbitrary aesthetic feelings or software limitations.

We will here present a systematic search for the optimum binning in the particular case of neutron reflectometry. This specific case is interesting per se because of the very large dynamic range of the data which poses particular problems. When planning the experiment, simulations of the expected data can help choosing optimal binning strategies and measurement time which will ensure getting the necessary information without grossly overestimating the needed data quality. This is especially important since low reflectivity measurements are in essence very time consuming and that one may wish to perform kinetic measurements as fast as possible.
Neutron Depth Profiling at the High Intensity Focused Beam of Reflectometer MARIAM

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Neutron depth profiling method (NDP) based on the energy analysis of charged particles produced upon the capture of thermal (cold) neutrons allows for the determination of the in-depth distribution of an appropriate elements (e.g. ⁶Li, ¹⁰B) with the depth resolution of 10-20 nm in a few micrometers thin layer of solids.

Some in situ (in operando) applications, e.g. the study of Li ions migration in all solid state thin film batteries, require very fast measurements. To fulfil this request, a new NDP setup is designed and constructed for the use at the extremely intense focused neutron beam of the reflectometer MARIAM (JCNS@MLZ). Being equipped with a multiple detectors for the collection of charged particles in a larger solid angle, this setup allows for unprecedentedly fast NDP measurements on the seconds scale without significant worsening of the depth resolution.
HERITAGE: the concept of a giant flux neutron reflectometer for the exploration of 3-d structure of liquid and solid interfaces in thin films.

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Keywords: neutron, instrumentation, reflectometry, GISANS, thin interfaces

The instrumental concept of HERITAGE [1] - a reflectometer with a horizontal sample geometry – well fitted to the long pulse structure of a neutron source is presented. It is constitutes a new class of reflectometers achieving the unprecedentedly high flux for classical specular reflectometry combined with off-specular reflectometry and grazing incidence small-angle scattering (GISANS), thus resulting in a complete 3-d exploration of lateral and in depth structures in thin films. This is achieved by specially designed neutron guides. In the horizontal direction (perpendicular to the scattering plane) the guides elliptic shape focusses the neutrons onto the sample. In the case of use of a compact high-brilliance flat moderator [2], a multichannel geometry in the vertical direction is foreseen to provide a smooth divergence distribution at the sample position while accepting the entire beam from the moderator.

The modular collimation setup of HERITAGE provides extremely high flexibility in respect to sample geometries and environments, including the possibility to study virtually all types of solid and liquid interfaces, statically or kinetically. The use of multiple beam illumination allows for reflectivity and GISANS measurements at liquid interfaces both from above and below without a need to move the sample.

This concept assures the delivery of the maximum possible and usable flux to the sample in both reflectivity and GISANS measurement regimes. Monte-Carlo simulations show that HERITAGE outperforms the flux of all present-day and already for the ESS planned reflectometers and GISANS setups in flux and in measuring time for standard samples. For example, the simulated averaged flux of HERITAGE for the ESS pancake moderator is \(7.6 \times 10^9\) n/cm²/sec. In comparison with the reflectometer design FREIA for ESS, the gain amounts to a factor of about 5 for 4x4cm² liquid samples and to factor about 8 for gain for 1x1cm² solid samples.

References:
Time-of-Flight and Monochromatic Polarized Neutron Reflectometry on D17 at ILL

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Keywords: instrumentation, reflectometry, polarized neutrons, magnetism,

We present recent upgrades of the polarized neutron reflectometer D17 at the Institut Laue-Langevin, Grenoble. Four modes of operation, time-of-flight and monochromatic, polarized and non-polarized, can be chosen from with minimal interference with the experimental configuration. This offers a data acquisition mode ideally suited for the experiment at hand, covering areas of materials, soft-condensed matter, biology and magnetic studies.

For polarized studies a monochromatic wavelength of \( \lambda = 5.5 \text{ Å} \) (\( \Delta \lambda / \lambda = 4\% \)) is delivered at a take-off angle of 4° with a polarization exceeding 98%. Alternatively, in time-of-flight mode, a recently commissioned S-Bender polarizing supermirror \( (m = 3.2) \) provides a wavelength band of 4 – 20 Å with flat polarization above 99 % (2 - 27 Å un-polarized). Polarization analysis is performed either using a supermirror or a polarized \(^3\text{He}-\text{cell} \) for off-specular scattering. Two radio-frequency spin-flippers with flat polarization efficiency of 99.5% for all wavelength provide access to all spin cross-sections.

A wide range of sample environment complements the suite of applications on D17, including cryogenic environments with large magnetic fields (7 T) in different directions. We will show experimental results highlighting the benefits of this advanced flexibility. The studies include magnetic gradients in thin film Heusler alloys, interfacial investigations on exchange-biased multilayers and off-specular scattering from lateral magnetic domain patterns.

We additionally introduce a *coherent* data reduction procedure\(^1\) allowing the use of fully divergent beams for optimized flux exploitation without resolution losses. The resulting gain enables measurements on sub-second time-scales. Similarly, non-flat samples, for example curved surfaces, can be analyzed with full flux and resolution recovery. The reduction procedure takes place in linear detector coordinates and can be directly implemented into existing procedures without the need for extra coordinate-transformations.

References:

Polarized neutron reflectometer NERO for reactor PIK

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Keywords: instrumentation, neutron reflectometry, neutron diffraction, polarized neutrons

The polarized neutron reflectometer with the polarization analysis NERO is designed as a multi-task reflectometer for the study of surface structures, interfaces, thin films, multilayer structures of both magnetic and non-magnetic materials.

In the framework of the Cooperation agreement between Petersburg Nuclear Physics Institute (Gatchina, Russia) and Helmholtz Zentrum Geesthacht (Geesthacht, Germany) NERO was transferred from the reactor FRG-1 to the new reactor PIK. The reconstruction work of the reflectometer is held in the framework of the project “Formation of the Gatchina-Geesthacht platform of neutron stations for research with neutron scattering techniques at the reactor PIK (PIK-GGBase)”.

The main characteristics of the polarized neutron reflectometer NERO are the following.

<table>
<thead>
<tr>
<th>Monochromator</th>
<th>HOPG (002), Δλ/λ = 0.02</th>
<th>Angular accuracy</th>
<th>0.001°</th>
</tr>
</thead>
<tbody>
<tr>
<td>Angular range</td>
<td>-20°&lt;20&lt;100°</td>
<td>The sample size</td>
<td>10×10 - 30×30 mm²</td>
</tr>
<tr>
<td>Horizontal collimation</td>
<td>0.01° - 0.05°</td>
<td>Detector</td>
<td>³He PSD with res. 256×256 pixels of 2×2 mm² pixel size</td>
</tr>
</tbody>
</table>

At the present the reflectometer is being upgraded. The first step of modernization was focused on the optimization the neutron guide system. NERO is being optimized for the wavelength of 5.18 Å and for resolution of 1%. We introduce a several modifications in the construction of this neutron instrument. We have provide a new supporting frame for beam former, collimation diaphragms and the first flipper. We have modernized a detector arm for a sample unit, for the second flipper, for the analyzer and, essentially, for the detector.

The new polarizing five channel system (so called the beam former) based on supermirrors Ni/Ti $m=2.5$ for unpolarized mode and CoFe/TiZr $m=2.5$ for the polarized mode replaces the old polarizing system, based on FeCo/Si supermirror. Its polarizing efficiency and transmittance of the beam are 0.98 and 0.80, respectively. Previous Mezei-type neutron spin-flipper (efficiency ≈ 96%) is replaced by the adiabatic radiofrequency flipper (with efficiency ≈ 99.9%). The new multichannel fan-analyzer on supermirrors CoFe/TiZr with $m = 2$ will be produced by PNPI NRC KI. The transmittance of the analyzer is of the order of 0.72.

The present work is carried out with the financial support of the Ministry of Education and Science of the Russian Federation, the Agreement on provision of grant No14.616.21.0004 from September 17, 2014, unique identifier of agreement RFMEFI61614X0004.
Monte-Carlo simulations for small angle neutron scattering instruments SANS-2 and SANS-3 at reactor PIK

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Keywords: neutron, instrumentation, Monte-Carlo simulations, neutron guide, small-angle scattering, polarization

The two small-angle neutron diffractometers SANS-2 and SANS-3 were transferred to Petersburg Nuclear Physics Institute of NRC “Kurchatov Institute” from Helmholtz Zentrum Geesthacht (HZG) and are to be optimized for operating at the high flux neutron source PIK when the reactor starts by the year 2019. The instrument SANS-2 was chosen to be adapted as a general purpose small-angle neutron scattering instrument due to its layout (18 + 22 m) and the existing polarization option. The instrument SANS-3 operated in Geesthacht as a polarized reflectometer PNR, has shorter layout (7 + 9 m) and is to be reconstructed in a non-polarized high flux SANS machine. Two individual neutron guides were optimized for both instrument using McStas ray-tracing package and the spectral parameters of the cold neutron source at the reactor PIK. Additionally, the loss mechanisms in neutron guides were simulated. The value of the flux at the sample position turned out to be few orders of magnitude higher than that at the HZG in the whole spectral range (4 – 25 Å). Finally, the new transmission polarizer for SANS-2 was optimized. These calculations are to become the basis for construction a set of demanded and competitive neutron instruments for condensed state studies at the reactor PIK.

The work was supported by Russian Federation Ministry of Education and Science (Federal target program, project №RFMEFI61614X0004).
Upgrade of the KWS-1 Small-Angle Neutron Scattering Instrument


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Keywords: KWS-1, SANS, polarized neutrons, polarization analysis

The KWS-1 small-angle neutron scattering (SANS) instrument operated by the Jülich Centre for Neutron Science (JCNS) at the research reactor FRM II of the Heinz Maier-Leibnitz Zentrum in Garching near Munich has been recently upgraded. Most of the parts of the instrument were installed for the first time, including: transmission supermirror polarizer, adiabatic radio-frequency spin flipper, chopper and neutron lenses. With the double-disc chopper, the wavelength spread can be reduced further from the standard 10% to about 1%. The lenses allow the enhancement of the neutron flux at the sample by using larger sample aperture. In this case the instrument resolution stays in the classical SANS range. The polarizer, with an average polarization > 93%, represents a three-channel V-cavity with Fe/Si coated supermirrors (m=3.6) and is positioned in a custom designed changer of revolver type. The flipper provides a high flipping efficiency of more than 99.9% for all neutron wavelength used at KWS-1. A custom designed hexapod in the sample position allows heavy loads and precise sample positioning in the beam for conventional SANS experiments as well as for grazing incidence SANS (GISANS) under applied magnetic field. A new flexible instrument control system (NICOS) is installed and its utilization opens new possibilities at the instrument.

The performance of the installed components was checked by means of the standard samples as well as 3He neutron spin filter in the case of polarizer and spin flipper characterization. Test measurements on a ferrofluid in a magnetic field with polarized and nonpolarized neutrons are presented. Recent developments in the field of polarization analysis at the instrument are presented along with the results of the first test.

References:
Small-angle neutron diffractometer SANS-2 for PIK Reactor

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Keywords: instrumentation, neutron diffraction, polarized neutrons, SANS

Small-angle diffractometer of polarized neutrons SANS-2 is designed for studies in the fields of materials science, the physics of metals, technology of nanostructures and nanomaterials, physics of the magnetic structures and superconductors, physics and chemistry of colloidal particles, microemulsions, colloidal solutions and liquid crystals.

In the framework of the Cooperation agreement between Petersburg Nuclear Physics Institute (Gatchina, Russia) and Helmholtz Zentrum Geesthacht (Geesthacht, Germany) the small-angle neutron diffractometer SANS-2 was transferred from the reactor FRG-1 (shut down in 2010) to the new reactor PIK (to be started in 2018). The reconstruction work to give a new life to SANS-2 at the reactor PIK had started in 2014 within the project “Formation of the Gatchina-Geesthacht platform of neutron stations for research with neutron scattering techniques at the reactor PIK (PIK-GGBase)”. The main characteristics of the small-angle diffractometer SANS-2 are the following. The monochromaticity of the neutrons beam is provided by the mechanical velocity selector that allows to run the instrument in the wavelength range from 4.5 to 20 Å with a resolution of Δλ/λ = 0.1. The collimation of the beam occurs in a collimation tube with a length of 18 m by round and rectangular apertures, with a maximum window size of 30×30 mm. The experiments can be performed with the polarized neutrons. The beam is polarized using a new compact transmission polarizer on silicon wafers with supermirror CoFe/TiZr (m = 2) coating. The polarizing efficiency and transmittance of the beam are 0.99 and 0.88, respectively. The adiabatic RF spin-flipper is installed to change the direction of the neutron spins with respect to the magnetic field. The guiding magnetic field with a value of 10 Oe is set along the entire collimation system using a system of coils. The sample area is equipped with an electromagnets and a refrigerator of the closed cycle that allows one to change the conditions on the sample in a wide range of fields and temperatures. The scattered neutrons arrive through the detector tube with length of 22 meters and are recorded by a two-coordinate position-sensitive detector with a size of 55×55 cm² with a resolution of 0.7×0.7 cm².

The assembly and check of the operation of the components and mechanisms of the small-angle diffractometer SANS-2 are currently carried out in the neutron-guide hall of the reactor PIK.

The present work is carried out with the financial support of the Ministry of Education and Science of the Russian Federation, the Agreement on provision of grant No14.616.21.0004 from September 17, 2014, unique identifier of agreement RFMEFI61614X0004.
SKADI – Small-Angle Neutron Scattering at ESS

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Keywords: neutron, instrumentation, European Spallation Source

The Small-K Advanced DIffractometer SKADI is a versatile SANS instrument, proposed for the European Spallation Source, which enables scientists to perform a wide range of investigations on topics requiring small Q-values to access long length scales. The scientific areas targeted by SKADI include investigations of smart materials, biological and medical research, magnetic materials and materials for energy storage, as well as experiments on nanomaterials and nanocomposites or colloidal systems. To maximize the applicability of these studies SKADI is designed to accommodate in-situ measurements with custom made sample environments to provide "real-world" conditions.

To achieve all these goals SKADI will feature the following general design properties:

- Flexibility (sample area is approx. 3x3 m², and versatile collimation)
- Very small Q accessible through VSANS (using focusing collimation elements)
- Polarization for magnetic samples and incoherent background subtraction
- Good wavelength resolution, being the longest SANS instrument
- High dynamic Q-range (using two detectors)

With a flux at about 25 times higher than at the D22, an accessible size regime between the Angstrom and micrometer scale and the high dynamic Q range for fast data acquisition with a high resolution both in Q and time, this instrument will open the ESS for a wide scientific community.

References:
Wide angle analyzer using On-beam SEOP polarized GE180 neutron spin filter cell

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Keywords: neutron, instrumentation, 3He neutron spin filters

Abstract: We present an ultra-compact ³He polarizer to be used as a polarization analyzer for separation of incoherent background for soft matter research. The ³He will be polarized in-situ within a very compact magnetic cavity, 18 cm long, that can be simply placed between the sample and the detector tank without lowering the maximum achievable Q range of the SANS machines we currently use at the JCNS. The angular coverage is a pyramid of 38° from the sample position. The optical pumping will be done with an ultra-compact volume Bragg grating frequency narrowed diode laser array bar. The full system will be readily hand transportable and thus useful as an instrument add-on. Additional, we present the progress of the fabrication of GE180 neutron spin filter cells that achieve both long relaxation times and high ³He polarization.
Novel type of polarisation analysis with the multianalyser at PUMA

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Keywords: Polarized neutrons, techniques and methods

Polarisation analysis is one of the most time consuming methods in neutron scattering. Part of this is the need to spend time for measuring the spin-flip and the non-spin-flip channels. By combining the multianalyser system at PUMA with an innovative polarization analysis setup saves beam time by allowing a simultaneous detection of both channels.

A combination of two polarisers/deflectors reflecting spin-down and transmitting spin-up neutrons are used to differentiate the neutron beam scattered by the sample. The separated beam components reach different blades of the PUMA multianalyser where they are reflected towards different detectors.

First test experiments performed recently demonstrated that the spin-up and spin-down components can be measured according to our expectations. The Bragg scattering of neutrons polarized by a $^3$He filter on Vanadium and an anti-ferromagnetic hematite ($\text{Fe}_2\text{O}_3$) sample were analyzed using the presented technique. In the case of the hematite nuclear reflex (006) only the transmitted (non-spin-flip) signal was recorded, whereas the anti-ferromagnetic scattering (003) shows both signals: transmitted (non-spin-flip) and reflected (spin-flip) signals. These two signals were clearly distinguishable with our setup and observed simultaneously in a single measurement. An energy scan with the vanadium sample demonstrated that polarisation analysis of the scattered neutrons can be successfully combined with the wavelength selection.

McStas simulations as well as analytical calculations were carried out to quantify the neutron distribution in the three resulting channels. The results of these two different approaches were compared to the results of the test experiment.
BIODIFF - Neutron Macromolecular Crystallography at the FRM II

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Keywords: neutron macromolecular crystallography, instrumentation, structure

The research reactor Heinz Maier-Leibnitz (FRM II) is a modern high flux neutron source which feeds at the present 27 state of the art instruments. The newly build neutron single crystal diffractometer BIODIFF is especially designed to collect data from crystals with large unit cells. The main field of application is the structure analysis of proteins, especially the determination of hydrogen atom positions. BIODIFF is a joint project of the Forschungszentrum Jülich (FZJ/JCNS) and the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II). Typical scientific questions addressed are the determination of protonation states of amino acid side chains in the active center of enzymes and the characterization of the hydrogen bond network between the protein and an inhibitor or substrate. BIODIFF is designed as a monochromatic instrument. By using a highly orientated pyrolytic graphite monochromator (PG002) the diffractometer is able to operate in the wavelength range of 2.4 Å to about 5.6 Å. Contaminations of higher order wavelengths are removed by a neutron velocity selector. To cover a large solid angle and thus to minimize the data collection time the main detector of BIODIFF consists of a neutron imaging plate system in a cylindrical geometry. A Li/ZnS scintillator CCD camera is available for additional detection abilities. The main advantage of BIODIFF is the possibility to adapt the wavelength to the size of the unit cell of the sample crystal while operating with a clean monochromatic beam that keeps the background level low. BIODFF is equipped with a standard Oxford Cryosystem “Cryostream 700+” which allows measurements in the temperature regime from 90K up to 500K.
Limitations on Displacement and Divergence of Longitudinal Neutron Resonance Spin Echo Spectroscopy

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Keywords: instrumentation, McStas simulations, divergence, neutron resonance spin echo spectroscopy

The beamline RESEDA (resonance spin echo for diverse applications)\textsuperscript{1} of the Heinz Maier-Leibnitz Zentrum (MLZ) has undergone a redesign from transversal to high-resolution longitudinal neutron resonance spin echo (LNRSE) spectroscopy. The change of the field geometry was accompanied by extensive McStas\textsuperscript{2} simulations and analytical calculations. McStas simulations reveal that already small rotational misalignments of the static $B_0$ Helmholtz coils suppress the polarization especially for high magnetic fields (see Fig. 1a) while translational displacements of the coils hardly affect it. Both deficiencies can easily be minimized by using standard rotation and translation tables. Furthermore, simulations show that the neutron flux density at the sample position can easily be increased by a factor of 10 by installing neutron guides (with $m = 1.2$) on the primary spectrometer arm of RESEDA. However, as the method is strongly dependent on the flight path lengths of the neutrons, beam divergence strongly affects the polarization. Detailed analytical calculations (see Fig. 1b) and McStas simulations taking into account the resonant spin flips show that for high $B_{pr}$-fields; i.e. large spin echo times, a small divergence is crucial.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig1.png}
\caption{a) Polarization as a function of the rotational displacement of the static $B_0$ coils for different field values\hspace{1cm}b) Dependence of the beam divergence on the polarization for different static $B_0$ field values.}
\end{figure}

References:


High-Resolution Neutron Spin-Echo Spectroscopy with Large Dynamic Range at RESEDA

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Keywords: instrumentation, neutron spin echo, spectroscopy, MIEZE, NRSE

The REsonance Spin Echo for Diverse Applications beamline RESEDA is a high resolution spectrometer with a large dynamic and momentum range.\textsuperscript{1} The instrument is located in the neutron guide hall West of the Heinz Maier-Leibnitz Zentrum (MLZ). RESEDA consists of one primary spectrometer arm and two different secondary spectrometer arms, schematically shown in Fig. 1. The instrument offers two dedicated spin echo options. The first option is high resolution longitudinal Neutron Resonance Spin Echo (NRSE) spectroscopy which gives access to a high dynamic and momentum range. In this technique, a pair of resonant flipper coils replaces the long solenoids which are used at classical Neutron Spin Echo (NSE) spectrometers. The second option is NRSE performing Modulation of IntENSITY with Zero Effort (MIEZE) which allows to investigate a large dynamic range of depolarizing samples and/or within depolarizing sample environments, because the beam polarization is exclusively manipulated along the primary spectrometer arm and; hence, before the sample position. Therefore, MIEZE allows investigating, e.g. ferromagnetic samples or samples in high magnetic fields as demonstrated by Kindervater et al.\textsuperscript{2}

We present the experimental setup of RESEDA, the NRSE and the MIEZE option in detail. The central topic is the spin manipulation and its technical realization. Moreover, exemplary scientific results provide a view on the versatility of the beamline and its applications.

Fig. 1: Schematic setup of the instrument RESEDA at the neutron guide NL5-S at the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II).

References:
Instrument and sample environment developments at Diffractometer STRESS-SPEC

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Keywords: STRESS-SPEC, materials science, sample environment

Abstract: The diffractometer STRESS-SPEC at the German neutron source Heinz Maier-Leibnitz (FRM II) is a dedicated instrument to tackle problems in the field of engineering and applied materials science. Modern materials science spans a wide field of scientific areas including the analysis of residual stresses, characterisation of textures, in-situ phase analysis of high performance alloys, and et al. [1, 2]. The different science topics require constant upgrade of the instrument setup and new sample environment options. In this contribution we will specifically review the recent installation of a new high resolution 2d-detector and show first results. In addition the extension of the already comprehensive special sample environment suite of STRESS-SPEC [3, 4] by a the specialized tensile/compression rigs for phase, strain and texture studies at elevated temperatures, as well as a newly configured dilatometer and its functionality will be presented.

References:

Neutron Stress-Diffractometer ARES for PIK Reactor

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Keywords: instrumentation, stress, strain, diffraction

The high penetration ability and relatively low absorption of neutrons make them a promising tool in non-destructive testing of bulk materials. Among other things, residual stresses formed in a material during manufacturing and/or various treatments can be easily measured by means of neutron diffraction.

Neutron diffractometry measurement of residual stresses is based on the same principle as convenient X-ray diffractometry and high-energetic synchrotron diffractometry. However, due to their very nature, X-ray diffraction is suitable for surface measurements, whereas synchrotron diffraction – for investigations of thin specimens or shallow depths within thick ones. In practice neutron diffraction is the sole non-destructive technique, which could reconstruct the whole 3D map of residual stresses inside a bulk detail.

The method consists in the measurement of shift of the position of the diffraction peaks from the positions defined by the unit cell parameters of the non-deformed material. It gives direct information about changing the interplanar distances, which can be easily converted into data on the long-ranged internal stresses, the so-called macrostresses. Analysis of the shape of the diffraction peaks can give information on the distortion of the crystal lattice within individual separated grains (microstresses).

In the framework of the “PIK-GGBase” project the neutron stress-diffractometer ARES was transferred from the closed FRG-1 reactor (Helmholtz-Zentrum Geesthacht, Germany) to the now under-constructed PIK reactor (Petersburg Nuclear Physics Institute, Gatchina, Russia). Briefly, the main parameters of the diffractometer ARES are as follows. The double focusing monochromator, based on elastically bent perfect Si crystals with reflecting plane (311), provides the neutron wavelength in the range of 0.16-0.23 nm, which is sufficient for the investigation of most commonly used crystalline materials. The specimen (weighing as much as 200 kg) could be scanned in 3 dimensions (x, y, z movements within range of 400 mm). Also the rotation around the vertical axis (omega-scanning) is provided. A wide choice of the gage volumes is supplied by the set of diaphragm slits, both for the incident beam as well as for the diffracted one. The He-3 area detector (300 mm x 300 mm) assures the uncertainty of the strain measurements of less than 0.01%.

During the last year the diffractometer is assembled and tested. The presented report demonstrates the characteristics of the instrument, the current state of the development, and required further modernization and improvements.

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New Sample Environments for Material Characterization at POWTEX Neutron Diffractometer, FRM II Garching

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Keywords: POWTEX, sample environments, in-situ diffraction

For deeper understanding of microscopic processes inside (geo)materials, scattering experiments under defined process parameters like temperature or pressure are necessary. Therefore, our group had developed sample environment equipment for the POWTEX (POWder and TEXture) Diffractometer located at the FRM II research reactor (Heinz Meier-Leibnitz Centre). This new diffractometer will offer unique possibilities for in-situ time-resolved texture and residual strain investigations during deformation and recrystallisation experiments, as the really high effective neutron flux and the large space for sample environments inside the detector system.

Therefore, it was possible to develop new important sample environments such as in-situ deformation apparatus for uniaxial and triaxial compressional deformation experiments in the neutron beam, which allow to investigate active deformation mechanisms by time-resolved measurements of the crystallographic preferred orientation (CPO). Furthermore, these apparatus can be used for the investigation of tensional stresses. They are designed for the investigation of either geological samples, which include the most relevant mineral phases of the earth’s crust and ice or material science sample material as well. Likewise, we have designed a rotatable laser-heated furnace to study in-situ recrystallisation processes in 3D by the stereological calculation of the measured textures¹. This furnace can be evacuated or used with reaction gases inside for chemical investigations.

To be able to measure texture and powder diffraction samples in a beamtime efficient way at POWTEX, we have also developed an automatic sample changer, which can be used for polycrystalline samples as well as powder samples in closed vanadium containers. This sample changer minimizes sample changing time extensively, as every sample change without the automatic sample changer is associated with an mechanical opening of the cylindrical detector prior to the hands-on sample changing routine. Furthermore, we have developed a Universal Stage which allows the easy and reproducible placement of sample environments like the laser furnace and magentcryostats in the neutron beam. This Universal Stage also allows to rotate sample environments around a vertical axis, if necessary.

References:
Field-Integral Homogeneity Optimisation Approach To The New Superconducting Solenoids For The Neutron Spin-Echo Spectrometer At MLZ

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Keywords: neutron, instrumentation, spin echo, dynamics

A semi-analytical method has been developed to optimise the magnetic fields inside cylindrical solenoids with minimal intrinsic field-integral inhomogeneity [1,2]. Following this approach a set of new superconducting solenoids has been designed for the upgrade of the neutron spin-echo spectrometer at MLZ (J-NSE). The new fully compensated solenoids will enhance the resolution of the spectrometer by reaching higher Fourier times. The lower intrinsic field inhomogeneity in the beam area will minimize the amount of required correction, which is currently difficult to improve with the existing Fresnel coils due to technical limitations. The new coils have been commissioned to Babcock Noell GmbH and will replace the currently used cylindrical copper coils. The new design will reduce the necessary corrector strength by over a factor 2 compared to present cylindrical coil designs and will provide field integrals up to 1.2 Tm.

The nanoworld in slow motion: scientific highlights and instrument developments at the J-NSE

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Keywords: neutron, instrumentation, dynamics, soft matter

The J-NSE neutron spin echo spectrometer faces now 10 years of successful user operation at the FRM II research reactor at the Heinz Maier-Leibnitz Zentrum (MLZ). We present scientific highlights and instrumental developments of the last decade, for example the development of grazing incidence neutron spin echo spectroscopy (GINSES) at the J-NSE and investigations of the dynamics at solid-liquid interfaces with this new option [1-3]. Polymers in confinement [4] have been a prominent topic, as well as the internal dynamics of proteins [5]. The scientific questions also triggered instrumental developments such as a new polarizer and a new neutron guide concept.

Finally, the future of the J-NSE will be addressed in a short presentation of the current upgrade program with superconducting main coils having a intrinsically reduced field inhomogeneity [6].

Comparison of neutron fluxes produced by proton and deuteron beams on a Be target: A planned experiment

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Keywords: neutron production, Be target, proton, deuteron

In the near future many research reactors in Europe used for neutron experiments will be shut down. The European Spallation Source (ESS), although very powerful, cannot fill this gap alone. Therefore, other facilities, like the proposed High Brilliance Neutron Source (HBS) [1], need to be built. In this type of facility neutrons are produced by bombarding a light Be-target with protons or deuterons with particle energies in the MeV range. In order to achieve a neutron flux comparable with modern medium-flux research reactors, the whole target-moderator system has to be optimized. The first decision is whether protons or deuterons shall be used as primary ions.

The neutron flux depends on the particle energy and the particle type due to different reaction channels for deuterons and protons. Here, the deuteron has additional reaction channels, like stripping and break-up, and should yield a higher neutron flux, which can be seen in MCNPX simulations using the TENDL nuclear data library [1]. But simulations with the TENDL database cannot reproduce the higher experimental neutron flux measured in [2]. In comparison to that, the ENDF database yields a higher neutron flux but is only available for protons.

Therefore, we are planning an experiment at the COSY accelerator at Forschungszentrum Jülich using the Mn bath technique following the setup described in [3] to determine the total neutron production rate. In this experiment we will compare the neutron fluxes after a bombardment of a thick Be-target with protons and deuterons with energies ranging from 5 MeV to 50 MeV. We will present the details of this experiment at the German Conference on Neutron Scattering in Kiel.

References:

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Abstract
The training and research reactor AKR-2 is a thermal, homogeneous, solid material moderated zero power reactor with maximum continuous power of 2 Watt. The AKR-2 was completely refurbished in 2005 and is currently the most advanced zero power training reactor in Germany.
The facility is equipped with a state-of-the-art digital I&C control system Teleperm XS (see also http://tu-dresden.de/mw/akr). The main purpose of AKR-2 and its design basis was and is the education of students in nuclear and reactor physics, in nuclear engineering as well as to teach fundamental knowledge and rules in radiation protection and radiation dosimetry. The structure of the reactor is shown in figure 1 below. The cylindrical core has a diameter of 250 mm and a critical height of 275 mm. The disk-shaped fuel elements consist of a homogeneous dispersion of polyethylene and uranium oxide (19.8 % enriched in Uranium-235). The core is completely surrounded by a graphite reflector. The neutron flux density at the core centre is about \(2.7 \times 10^7\) n/(cm\(^2\)-s). The neutron spectrum at different local positions is shown in figure 1.

Due to the physical characteristics of AKR-2, research is limited to projects where low neutron fluxes are desirable and variable operational conditions as well as low costs are requested. The access to AKR-2 is uncomplicated and there is no proposal reviewing system. While at high flux facilities high level and well recognized research is carried out, AKR-2 is ideal for idea, test and quick trial experiments. There are a couple of experimental channels enabling flexible access to the neutron field. The coupled neutron – gamma field at defined points within and in the neighborhood of the multiplying medium is well characterized which makes the AKR-2 attractive for some specific research topics. Examples are investigations on sophisticated neutron detectors, development of radiation measuring techniques, radiation spectrometry in mixed neutron-photon fields and validation of reactor physics codes. In the presentation, we will give a detailed description of the AKR-2’s design and its physical properties. Furthermore, we would like to discuss the question how the AKR-2 can contribute in neutron science in future?
Low dimensional thermal and cold finger moderator for the High Brilliance Neutron Source Jülich

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Keywords: HBS, neutron source, instrumentation, CANS, moderator

Abstract

For the future accelerator based neutron source project HBS at Forschungszentrum Jülich1 we plan to use the nuclear Be(p,n) or Be(d,n) reaction with accelerated particles in the lower MeV energy range. We intend to compensate the lower “produced neutron per incident particle” ratio, compared to spallation, by improving the neutron extraction process and optimizing the brilliance at the sample. The crucial role of extracting thermal and cold neutrons directly from the neutron density maximum within the thermal moderator will be achieved by the novel finger moderator, whereas the geometric and moderating properties of the finger are optimized towards brilliance according to the principle of low dimensional moderators proposed for the construction of the ESS “Butterfly” moderator3. Experiments to verify the MCNP model calculations2 have been conducted with a D2O moderating reflector prototype feeding an optimized cold moderator at the AKR-2 reactor in Dresden, which with the nominal power of 2W provides a source strength of 10^8 n/s with the fission spectrum on the inner moderator surface. Thermal and cold beam extraction from the flux maximum within the moderator based on liquid H2 and other moderators like solid methane and mesithylene will be tested by energy spectroscopy via TOF-method. Different liquid H2 ortho/para ratios will be investigated and controlled in real time via online heat capacity measurements to be able to modify the cold neutron spectrum to instrument needs.

References:

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Neutron guide H3-2 at the high-flux reactor PIK

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Keywords: neutron, instrumentation, neutron guides

The start of the high-flux PIK reactor hosted by Petersburg Nuclear Physics Institute (PNPI NRC “Kurchatov Institute”), Gatchina, Russia, is foreseen in 2019. Having thermal power of 100 MWt PIK reactor will become the most powerful steady state neutron source in the world. Initially the reactor will be equipped with one cold source which will feed neutrons to the large neutron guide hall via the extensive neutron guide system.

The Phase I instrument suite includes several instruments to be placed in the neutron guide hall: two small-angle neutron scattering (SANS) instruments, two Ultra SANS instruments, and two neutron reflectometers. Part of them (namely SANS-2, SANS-3, DCD and NeRo) were transferred to PNPI from Helmholtz Zentrum Geesthacht (HZG) and are currently undergoing deep modernization.

Here we describe the neutron guide H3-2 dedicated for ultra-small angle instrument DCD, reflectometer NeRo, vertical scattering plane reflectometer REVERANS and the cold neutron powder diffractometer. Main features of this neutron guide include: the initial expanding part to increase cross-section and to lower divergence, the individual branches for each instrument; the full optical optimization to satisfy instrumental needs; the elimination of the direct line-of-sight inside the bunker more than once. All guide branches were simulated using Monte-Carlo technique. The overview of the final design will be given along with the beam properties at the guide exits and the sample positions.

The search for the best guide geometry for this suite made us conduct an additional study of the joint optimization of the neutron guide and focusing monochromator. In addition to the straight guide, the elliptical and parabolically expanding ones were studied in order to deliver the maximal flux at the sample position.

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E- Neutrons - The E-Learning Platform for Neutron Scattering

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Keywords: e-learning, simulation

Introduction: Since experimental neutron scattering is mostly restricted to large-scale facilities, not all students have access to learning the technique at their home institution. Providing a freely accessible e-learning portal for neutron scattering is therefore an important outreach task in order to secure and educate the future users and scientists at neutron scattering facilities.

The Platform: To address this teaching challenge, the partners have developed and utilized an e-learning portal for neutron scattering under the working title “Virtual Neutrons for Teaching” (VNT) – now fully developed with frontpage available at http://www.e-neutrons.org. - See Figure 1.

Once a user is registered at e-neutrons.org, access is granted to the full e-learning platform, made up from three main parts:

1. A “WIKIbook” provided by Mediawiki with various extensions, e.g. for producing mathematical expressions, playing the role of textbook-material
2. A Learning Management System (LMS) provided by the Moodle system, providing exercises, quizzes and evaluation
3. A web simulator for the McStas neutron instrument simulation program, allowing to perform virtual neutron scattering experiments.

Learning material: 10 neutron scattering modules have been designed, whereof 8 are general introduction to neutron scattering and techniques and 2 are specialised applications in imaging and magnetic neutron scattering. Two courses are constructed from these modules, differing in learning path restrictions and guidance. The courses contain a variety of student activities such as learning quizzes with hints and immediate feedback and exercises with hints and solutions. Using the online simulator that contains virtual instruments with samples, the student can tune pre-defined parameters to obtain synthetic data from Monte-Carlo simulations. The resulting data can be downloaded and analysed like real, measured data. In quiz-lessons connected with some of the instruments the students can explore through theory and virtual experiments, how a neutron scattering technique is used to solve a scientific problem.

Acknowledgement: The main funding for the project is provided by the European Union’s 7th Framework Program for research, technological development and demonstration under the NMI3-II Grant number 283883 and from the European Union’s Horizon 2020 research and innovation program under grant agreement No 654000.
Davinci: A Software for the Visualization and Processing of Single-Crystal Diffraction Data Measured with a Point Detector

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Keywords: data reduction, data visualization, single-crystal diffraction

Although area detectors have become standard equipment for X-ray diffractometers at synchrotron radiation sources, this is not the case for the neutron instruments with short wavelengths. For instance, two hot-neutron diffractometers HEiDi and POLI at MLZ are currently using only point detectors. As such detectors provide often more accuracy to measured intensities they are still in use at some other neutron facilities if high precision in the determination of crystal and magnetic structures is essential. Data reduction software for neutron diffractometers with point detectors is often instrument-specific tool related to its geometry and measured data format. For instance, the data collected using the recently commissioned setup of the new instrument POLI cannot be directly treated by the data reduction program Pron available on HEiDi. This contribution presents Davinci¹, a new software for the visualization and processing of single-crystal diffraction data measured with a point detector. Davinci allows to extract the integrated intensities of the Bragg reflections from the raw data points of measured scans for further analysis (with FullProf, Jana2006, ShelX, etc.). Recent studies of the multiferroic melilite Ba₂CoGe₂O₇ are based on the data obtained using Davinci². In addition to the conventional and Lehmann-Larsen algorithms for the definition of the peak and background parameters (also available in Pron), Davinci offers least-square peak fitting routines as well as a procedure to eliminate the shoulders from neighbored peaks. With this new software, the treatment of both the whole data set and individual peaks is noticeably simplified due to the convenient and intuitive graphical user interface (Fig. 1). Davinci is developed in C++ using the modern multi-platform Qt framework. Currently, it supports data collected at POLI, HEiDi and MIRA either with their own instrument operation programs or by the MLZ standard instrument control system NICOS. Support of the data format from instruments at other neutron facilities is requested and planned (6T2 at LLB and TRICS at PSI).

References:
BornAgain: Simulate and Fit Grazing Incidence Small Angle Scattering

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Keywords: GISAS, software, simulation, DWBA

BornAgain [1] is a multi-platform open-source project that aims at supporting scientists in the analysis and fitting of their GISAS data, from both synchrotron (GISAXS) and neutron (GISANS) facilities. The software provides a generic framework for modeling multilayer samples with smooth or rough interfaces and with various types of embedded nanoparticles.

BornAgain software is provided with a graphical user interface as well as a Python API. The sample model includes over twenty particle form factors, particle size distribution and complex composite particles as well as roughness of the layers. The instrument model accounts for the resolution effects and two kinds of detectors: rectangular and spherical. The simulation of the scattering process is performed in the framework of the distorted-wave Born approximation (DWBA). Both polarized and unpolarized neutron scattering can be simulated.

References:
Refining angular- and wavelength-dispersive neutron time-of-flight powder-diffraction data

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Keywords: neutron, instrumentation, structure, dynamics

We present an extension of the well-established Rietveld refinement method for modeling two-dimensional neutron time-of-flight powder-diffraction data, i.e., intensity as function of diffraction angle $2\theta$ and wavelength $\lambda$. For the future instrument POWTEX [1] at FRM II and also for the future powder diffractometer DREAM at the ESS, this novel approach will fully exploit the varying resolution with $2\theta$ and $\lambda$ which is otherwise lost when integrating with today’s standard refinement procedures.

For a proof-of-concept, powder diffraction data of CuNCN and diamond was collected at POWGEN, SNS, Oak Ridge. Major steps of this new analysis are as follows. Using a modified reduction procedure the POWGEN event data is binned logarithmically in $d$ and linearly in $d_\perp$, a newly introduced variable. Together they span an alternative orthogonal variable space [2]. The 2D (dimensional) pattern is corrected for 2D-background and calibrated by a 2D-vanadium pattern to account for detector efficiency and the wavelength-dependent intensity distribution. A suitable 2D analytical profile function describing the instrument characteristics needs to be established for the data refinement. Therefore, the peak profile as a function of $2\theta$ and $\lambda$ is determined from a reference samples and provided to the user as an instrumental resolution file.

Finally, we aim to implement the multi-dimensional refinement procedure into common Rietveld program suites in order to allow the users all necessary data-treatment possibilities. This new development is open for joint activities with program authors of existing user software, and we welcome current considerations towards an implementation into GSAS II.


A New Multi-MHz Detection System Operational at KWS-2 High-Intensity SANS Diffractometer of the JCNS at MLZ

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Keywords: SANS, neutron detectors, structure

The small-angle neutron diffractometer KWS-2, operated by the Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), is dedicated to the investigation of mesoscopic multi-scale structures and structural changes due to rapid kinetic processes in soft condensed matter and biophysical systems. Following demands from the user community, it was recently considerably upgraded [1], aiming for boosting its performance with respect to the intensity on the sample, the instrumental resolution, and the minimum scattering variable $Q_{\text{min}}$.

A vast performance improvement was further achieved by the recent upgrade of the instrument’s main detector. A new detection system has been newly designed by GE Reuter Stokes for KWS-2, to meet requirements related primarily to the high count rates available, in the MHz range, and high pixel rate stability. The system consists of an array of 144 $^3$He tubes with an active detection area equivalent to 0.9 m² (covering entirely the cross-section of the 1.4 m detector tank) and an innovative rapid readout electronics. The $^3$He tubes (with a global efficiency per tube of 85% for $\lambda = 5$ Å) are grouped in eighteen 8-pack modules comprising, different tube lengths, that work in parallel, each module having its own processor and electronics. To improve the readout characteristics and reduce the noise, the detection electronics is mounted in a closed case on the backside of the $^3$He tubes frame. The performance of one 8-pack module and of one single tube detector, in terms of count rate, resolution, pixel rate stability and position stability, was tested beforehand in high flux conditions at KWS-2, while the performance of the whole detection system was assessed during the detector commissioning phase at KWS-2, in the fall of 2015. A resolution better than 8nm and a count rate of about 90 kHz per tube (for the 820mm medium long tubes) at 10% dead-time for a homogeneous illumination were measured. The new detection system is characterized by an effective dead-time constant of 25 ns and an overall count rate as high as 5 MHz at 10% dead-time for flat profiles, which is an improvement of factor 25 compared to the old detector. These features are due to the fact that the system contains independent channels operated in parallel, which is an advantage over systems that experience dead-time after one event. The much higher count rate will shorten the measurement times and thus, increase the number of experiments in the same time period by the optimal use of the high flux of up to $2 \times 10^8$ neutrons cm$^{-2}$ s$^{-1}$ at the sample position. In addition, this will enable new scientific opportunities in the field of the structural investigations of small soft-matter and biological systems. These systems typically deliver at high wave-vector transfers $Q$ only a very weak scattering signal above the buffer or solvent level, which now can be resolved due to the performance jump enabled by the new detector.

Here, we present in details the concept and characteristics of the new detection system and its integration and commissioning at KWS-2, supported by results obtained during the performance characterization tests and early routine measurements.

References:

SOLID-STATE NEUTRON DETECTOR SoNDe - A NEW NEUTRON DETECTOR FOR HIGH-FLUX APPLICATIONS

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Keywords: neutron, instrumentation, detectors, scintillation

The Solid-State Neutron Detector – SoNDe – project aims to develop a high-resolution neutron detector technique that will enable the construction of position-sensitive neutron detectors for high-flux sources, such as the upcoming European Spallation Source (ESS). This includes also the construction of a full-scale prototype as a research and innovation action. Moreover, by avoiding the use of 3He in this detector the 3He-shortage, which might otherwise impede the construction of such large-scale facilities, can be alleviated.

The main features of the envisioned detector technique are:

- high-flux capability, capable of handling the peak-flux of up-to-date spallation sources (gain factor of 20 over current detectors)
- high-resolution of 3 mm by single-pixel technique, below by interpolation
- high detection efficiency of 80 % or more
- no beam stop necessary, thus enabling investigations with direct beam intensity
- strategic independence of 3He
- time-of-flight (TOF) capability, necessary to exploit maximum flux, with a time resolution in the µs regime
- modularity, improving maintenance characteristics of today’s neutron detectors

Compared to nowadays detector techniques a gain factor in counting rate of 20 (for 3He detectors) is possible. Such gain factors will be needed to make the best possible use of sources such as the European Spallation Source (Sweden), the Institut Laue-Langevin (France) and the Maier-Leibnitz Zentrum (Germany). Benefiting instruments at such a facility, among others, would be Small-Angle Neutron Scattering (SANS) instruments such as SKADI,1 but also reflectometers or any other instrument with a need of high-flux capability and a mm resolution. Also applications outside neutron scattering, for example positron emission tomography, are conceivable.

In this presentation first results about a SoNDe technical demonstrator will be shown, where countrates up to 125 kHz on a 5x5 cm² detector and a radiation hardness of 10 years anticipated ESS operation were achieved.

References:
The CASCADE Project - On the Phase Front of Neutron Detection

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Keywords: thermal neutron, detector, spin echo, position sensitive, instrumentation

The world of detectors used in neutron scattering instrumentation has changed. Much of what once was established has been discarded. By alerts on the future Helium supply critical to perspectives of the European Spallation Source, the run on substitutional technologies started. We report on the CASCADE Project [1] - a novel detection system, which has been developed for the purposes of neutron spin echo spectroscopy. It features 2D spatially resolved detection of thermal neutrons at high rates. The CASCADE detector is composed of a stack of solid Boron-10 coated Gas Electron Multiplier (GEM) foils, which serve both as a neutron converter and as an amplifier for the primary ionization deposited in the standard counting gas environment. This multi-layer setup increases the detection efficiency of a single entity. For the application in MIEZE spin echo techniques, which make use of the coherence of fast oscillating neutron interference patterns, the signal of the charge traversing the stack is detected to identify the very thin conversion layer of about 1 μm. This allows to precisely determine the time-of-flight [2]. Here we literally sit on the phase front of neutron detection. The RESEDA and MIRA Spectrometers at the FRM II run such new generation systems. This talk will discuss the characteristics of the system, challenges and perspectives, explicitly on the basis of the CASCADE detector at RESEDA.

References:

Figure: Phases of the neutron beam separated by GEM layer (left to right equals front to back in beam direction). This MIEZE measurement at RESEDA was conducted at a frequency of 53.5 kHz and a wavelength of 8.05 Å. The color code scales half a period from 0 to π, which equals 9.34 μs. The inner layers are approximately half a period in distance.
Chopper System of the Beamline for European Materials Engineering Research (BEER) at ESS

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Keywords: neutrons, instrumentation, engineering

Fundamental objectives of materials science are the development of new materials, their characterization and studies of their microstructure–properties relationship. One of the most used tools to achieve these goals is neutron time-of-flight diffraction in combination with in situ experiments. However, the modern materials are often complex and require novel techniques, allowing investigations under real working conditions and on realistic time scales. Thus, high time and spatial resolution even with small samples volumes are urgently required by a multidisciplinary community of scientists. For this purpose, HZG and NPI are proposing to build the engineering diffractometer BEER – Beamline for European Materials Engineering Research – at the European Spallation Source (ESS). The ambition is to serve the growing community of materials scientists with a sophisticated diffractometer, unique by its novel pulse multiplexing technique based on extracting several short pulses out of one long ESS pulse. Multiple short pulses enable diffraction with high resolution and high intensity.

Monte Carlo simulations have been performed using the McStas software package in order to optimize the beamline. They show that BEER will outperform existing diffractometers for the case of high-resolution diffractometry e.g. for strain analysis in highly symmetric materials. Furthermore, BEER will offer the opportunity of combining small-angle neutron scattering and neutron imaging investigations with high performance in a single instrument without compromising the diffraction performance.
**T-REX: Time-of-flight Reciprocal space Explorer, the bispectral direct geometry chopper spectrometer at the ESS**

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Keywords: neutron, instrumentation, dynamics

We present the concept of the bispectral direct geometry chopper spectrometer for the ESS neutron source. The instrument is designed to yield a dynamic range that extends from 20 µeV to 140 meV in energy transfer and from 0.01 Å\(^{-1}\) to 17 Å\(^{-1}\) in wavevector transfer. The elastic energy resolution (FWHM) can be freely adjusted in the range from 1% to 3% at 3 meV and from 4% to 8% at 100 meV.

T-REX has been benchmarked against existing state-of-the-art neutron TOF-spectrometers and shows flux gain factors between one and two orders of magnitude and possible solutions are foreseen to increase the signal to noise ratio. These features make the instrument a real game changer in wide areas of scientific research: magnetism, strongly correlated electron materials, functional materials, soft-matter, biophysics and disordered systems. Independent of the specific application, it will allow parametric studies with acquisition times below one hour for a full data set from single crystals, the application of extreme conditions, in-situ or in-operando studies on timescales of seconds or spectroscopy from sub 100 mg single crystals.

It will implement time-of-flight spectroscopy with Polarization Analysis as a standard tool, e.g. for studying the effect of confinement on the magnetic excitations in nano-particles or to uniquely derive the vibrational hydrogen excitations in soft matter through separation of the nuclear spin incoherent scattering.
The Concept and Capabilities of the DREAM Diffractometer at ESS

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Keywords: neutron diffraction, instrumentation, 2D Rietveld analysis, European Spallation Source

Neutron diffraction instruments are the workhorses of any large-scale neutron scattering facility and are among the first instruments to be enrolled into a user program. Here, we present the concept of the Diffraction Resolved by Energy and Angle Measurements (DREAM) powder diffractometer which is going to be built at the European Spallation Source (ESS) as a German in-kind contribution. The design of DREAM utilizes the long pulse and unprecedented peak brightness of ESS to provide a flexible choice between the high resolution and high intensity. We note that the estimated highest resolution in the backscattering detector is $\Delta d \sim 0.00028 \, \text{Å}$, which will set a new world record in neutron diffraction. The instrument is about 75 m long, facing both thermal and cold moderators. A solid Si bender will be reflecting the cold neutrons into the incident beam, while transmitting the thermal neutrons. As a result, the neutron bandwidth will include both thermal and cold neutrons at their peak intensities, providing the $Q$-range of $0.01 - 25 \, \text{Å}^{-1}$ in a single frame, which is ideally suited for diffraction studies on multiple length scales. The pulse shaping chopper consisting of two fast counter-rotating disks will allow a seamless change between the high resolution and high intensity modes of operation. Additional overlap, $T_0$ and band control choppers are going to be installed to reduce the instrument background. The ballistic neutron guide with elliptical end sections is optimized for a high brilliance transfer and it is particularly tailored towards the transport of the short wavelength neutrons. The new 2D position sensitive $^{10}$B detectors cover about 6 sr. and enable neutron time-of-flight Laue single-crystal diffraction measurements. Finally, our simulations indicated that DREAM will outperform the existing instruments at other facilities and provide unmatched flexibility in resolution and intensity. A full potential of DREAM will be realized through a new approach to the data analysis, which is based on 2D Rietveld refinements of the angular- and wavelength-dispersive diffraction data. The DREAM instrument will outreach the large scientific communities in material science, solid state physics, chemistry, magnetism and nanoscience.

References:
The High Flux Backscattering Spectrometer IN16B at ILL

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Keywords: Instrumentation, Neutron backscattering

The new high flux backscattering spectrometer IN16B at the Institut Laue-Langevin (ILL) offers in its standard configuration with strained single crystal Si111-monochromator and Si111-analysers a sub-µeV energy resolution of ~0.75 µeV within a dynamic range of ±31 µeV and simultaneously in a wide momentum transfer region (0.2<Q<1.8 Å⁻¹). This most advanced reactor backscattering instrument surpasses the old IN16 by more than one order of magnitude in count rate, a tripled signal-to-noise ratio and a doubled dynamic range. This was achieved by a consequent layout of the neutron optics, starting with the sample and the Doppler monochromator, the acceptance range of which is favorably covered by neutrons arriving from a Phase-Space-Transformation (PST) chopper, where its own ‘useful’ phase space region is in turn served optimally by a ballistic neutron focusing guide with velocity selector. Other improvements came by the increase of the analyser space angle, a vacuum chamber and background shielding including a background chopper. IN16B is a versatile instrument with several configurations: thus its resolution can be improved by ‘polished’ unstrained Si111 crystals to about 0.3 - 0.4 µeV or in long terms possibly even to 50 neV using GaAs200 crystals – an ongoing project in collaboration with FAU Erlangen financed by the BMBF will show its feasibility. The instrument can also be operated with the Si311-configuration to reach large Q (0.7 < Q < 3.5 Å⁻¹ with 2 µeV resolution within ±59 µeV; commissioned 2015) and from next year on with a time-of-flight option (BATS), which will extend the dynamic range to ±250 µeV – again in collaboration with FAU Erlangen and financed by the BMBF. The high beam time request for IN16B shows the large interest in its outstanding performance and new possibilities with inelastic fixed window scans. Publications in a wide range of science are starting to appear.

References:
[3] K. Kuhlmann et al., contribution at this meeting

Fig.1: The high flux backscattering spectrometer IN16B at ILL in its ‘High Flux Position’ (l.h.s.) and its new analysers (r.h.s.)
Pushing The Limits Of Neutron Backscattering: A GaAs Option For IN16B At ILL

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Keywords: Instrumentation, Neutron Backscattering, Spectroscopy

We present the ongoing "GaAs" extension project for the backscattering spectrometer IN16B at ILL, comprised of a prototype monochromator and analyser (total crystal surface area: \(\sim 1 \text{ m}^2\), to be installed in autumn 2016) with an energy resolution below 50 neV FWHM, a maximum energy transfer range of \(\pm 5 \mu\text{eV}\), and covering a Q-range from 0.2 to 2 Å\(^{-1}\).

This opportunity to enhance the energy resolution is provided by the relative extinction width of the GaAs 200 reflection \(\Delta k/k = 1.57 \times 10^{-6}\), corresponding to an energetic Darwin plateau width of \(\Delta E = 8.04 \text{ neV}\), which is about one order of magnitude smaller than that of the currently utilised Si 111 with \(\Delta k/k = 18.6 \times 10^{-6}\) and \(\Delta E = 77 \text{ neV}\)\textsuperscript{1,2}. Using a two crystal test setup on IN10 at ILL, neutron measurements of commercial VGF GaAs wafers, structurally strengthened by Si/B doping, yield convolved line widths of 17–28 neV FWHM, confirming the possibility to significantly improve the energy resolution of a backscattering spectrometer.

To keep the divergence term sufficiently small, the crystal surface area of \(\sim 1 \text{ m}^2\) will be built out of 4 \(\times\) 4 mm\(^2\) squares with a misalignment \(\Delta \theta < 0.1^\circ\), and variations of the lattice parameter related to fluctuating dopant concentrations either between different crystals or during the growth in one crystal must not exceed a value of \(\Delta a/a \sim 10^{-6}\). Further, the crystal temperature will have to be actively controlled with an accuracy of \(\Delta T \sim 0.1 \text{ K}\). This feature will be used to compensate for the energy shift of 103 neV/m induced by gravity at the tall analysers with a height of 3 m, requiring a difference of 10 K between top and bottom.

References:
SANS Study of Vortex Lattice Structural Transition in Optimally Doped Ba$_{1-x}$K$_x$Fe$_2$As$_2$

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Keywords: iron-based superconductors, vortex lattice, second magnetization peak

We have used small-angle neutron scattering to study the vortex lattice (VL) in high quality optimally doped (Ba$_{1-x}$K$_x$)Fe$_2$As$_2$ single crystals. Where previous SANS studies, as well as real-space imaging methods applied to the study of the vortex ensemble in single crystalline (Ba$_{1-x}$K$_x$)Fe$_2$As$_2$, Co-, and P-substituted BaFe$_2$As$_2$, have consistently reported highly disordered vortex structures [1]-[7], the present SANS study reveals sharp vortex lattice Bragg peaks. This is the first iron-based superconductor, apart from the "clean" end-compound KFe$_2$As$_2$ [8] in which an triangular VL is observed. In this contribution we present SANS data taken in a magnetic field ranging between 0.25 and 2 Tesla, performed on optimally doped (Ba$_{1-x}$K$_x$)Fe$_2$As$_2$. The data show clear resolution-limited sharp Bragg spots, indicating the existence of a long-range ordered Bragg glass in this compound. The magnetic field dependence of the vortex structure factor, obtained by correction of the intensity by the field-dependent vortex form factor, shows a sharp drop well below the second critical field. This vortex structural transition shows clear correlations with features observed around the so-called "second magnetization peak" observed in isothermal hysteresis loop measurements, and the behavior of magnetic hysteresis between zero-field cooling and field-cooling.

References
A novel hybridized crystal field - phonon excitation in the non-centrosymmetric heavy fermion compound CeAuAl₃

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Keywords: strongly correlated electron systems, crystal electric field, phonons

Hybridized excitations that comprise of well-understood collective modes have received increasing interest as the possible origin of unconventional materials properties and novel functionalities. In strongly correlated electron systems the effects of electron-phonon interactions are typically neglected, being deemed not important for an overall understanding. However, recently neutron time-of-flight spectroscopy on polycrystalline CeCuAl₃ have provided putative evidence for a vibron, i.e., a combined crystal field – optical phonon excitation [1], whereas no such excitation could be detected in the isostructural sister compound CeAuAl₃ [2]. This raises the question to what extent such hybrid modes represent a generic property of the series of Ce₇Al₃ compounds (T: transition metal element) or even f-electron systems in general. To pursue this question, we have revisited the properties of CeAuAl₃ using triple axis neutron spectroscopy on a float-zoned high-quality single-crystal. In contrast with early conjectures, we find two pieces of strong evidence suggesting strong crystal field – phonon interactions and the formation of a novel hybrid mode. First, at the zone center there is clearly a hybridized excitation between the crystal-field and phonons, which appears to be in general agreement with vibronic bound state reported for CeCuAl₃ [1]. However, in our single-crystal study of CeAuAl₃ we can clearly attribute this mode to the interaction of the crystal field with acoustic phonons at the zone boundary. Second, we observe a distinct anticrossing of the longitudinal acoustic (LA) phonon with the Γ₇(1) crystal field level (see Fig. 1). To the best of our knowledge such an anti-crossing has not been reported before. Both phenomena are in agreement with observed dominant phonon scattering processes by the localized 4f electrons [3]. Taken together, our results suggest that strongly hybridized crystal field – phonon excitations may, in fact, be rather common in f-electron compounds.

References:

Investigation of Magnetic Correlations in Nanoparticle Supercrystals Using Small Angle Neutron Scattering

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Keywords: magnetic nanoparticles, small angle scattering, dipolar interactions, magnetic correlations

Self-assembly of nanoparticles constitutes a novel approach to fabricate magnetic nanostructures. These structures form a new type of artificial material, in which the nanoparticles take over the role of atoms in conventional materials. While the magnetic properties of individual or weakly interacting magnetic particles are not quite understood, this is even more the case for the collective magnetic behavior in dense nanoparticle supercrystals. We have used centrifuge assisted sedimentation to fabricate very large 3D nanoparticle supercrystals (i.e. nanoparticle 'macrocrysats') from spherical iron oxide nanoparticles with a diameter of 15 nm. The assembly of macrocrystals up to 300 \( \mu \text{m} \) in size was possible. The magnetic behavior was investigated using magnetometry as well as semi-polarized small angle neutron scattering performed on MARIA, the high intensity reflectometer at the Heinz Maier-Leibnitz Zentrum (MLZ). As the samples were still very small, the neutron experiment proved to be challenging. Nevertheless, the magnetic ordering in dependence of an external magnetic field is clearly observed as shown in figure 1.

Figure 1: Semi-polarized SANS plots in transmission through a nanoparticle supercrystal as difference between the spin-up and spin-down channel for two external magnetic fields. The visible shadow is an artifact of the beam stop.
Magnetic excitations in quantum-critical Ce(Pd$_{0.86}$Ni$_{0.14}$)Al

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Keywords: neutron, magnetism, heavy-fermion compounds, spin dynamics

The heavy-fermion compound CePdAl offers the opportunity to study quantum criticality in the presence of geometrical frustration. The frustration is a consequence of the cerium atoms located on a distorted Kagome lattice in the hexagonal basal plane. The antiferromagnetic order in CePdAl with $T_N = 2.7$ K decreases strongly when substituting nickel for palladium and a quantum critical point with $T_N = 0$ is approached in CePd$_{1-x}$Ni$_x$Al for $x = 0.14$. We performed extensive inelastic neutron scattering on the cold triple axis spectrometer IN12 at ILL to study the low-energy magnetic excitations in single-crystalline Ce(Pd$_{0.86}$Ni$_{0.14}$)Al, i.e., the alloy where magnetic order is just suppressed to zero. We find spin fluctuations being broad in q-space indicating only short-range dynamical spin correlations. However, they display an almost critical slowing down as expected for a system close to an ordered state. We discuss the magnetic response in Ce(Pd$_{0.86}$Ni$_{0.14}$)Al, its temperature and magnetic field dependence and will compare the results to the spin excitations in the magnetically ordered parent compound CePdAl.
Magnetic Frustration and Domains in Metallic CePdAl

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Keywords: neutron, magnetism, frustration, domains, heavy-fermions

Magnetic frustration and its effects on magnetic properties of materials are intensively investigated in the field of condensed-matter physics. Novel states of matter, which are characterized by strong correlations, e.g. spin liquid or spin ice states, were observed in many electrical insulators [1]. CePdAl might be a promising candidate to study the effects of magnetic frustration in a metallic heavy-fermion system. The presence of the Kondo effect, RKKY interaction and magnetic frustration results in a complex interplay of competing interactions in this material. Thermodynamic and resistivity measurements indicate a Kondo temperature of \( T_K = 5 \text{ K} \) in CePdAl [2,3]. It orders antiferromagnetically below a Néel temperature of \( T_N = 2.7 \text{ K} \) and reveals a strong magnetic anisotropy between the \( c \)-axis and the \( ab \)-plane (susceptibility ratio around \( T_N \) is roughly 19) [4]. Due to its hexagonal crystal structure, the magnetic Ce\(^{3+} \) ions are arranged on a distorted Kagomé lattice in the basal plane [5]. The existence of edge-sharing triangles and the strong Ising character of CePdAl leads to magnetic frustration. Early neutron scattering experiments on powder revealed, that only two thirds of the Ce moments participate in long-range magnetic order, whereas the other third remains mainly disordered [5]. The propagation vector was found to be \( \mathbf{q} = (0.5 \ 0 \ \tau) \), where \( \tau \approx 0.35 \) is slightly temperature dependent above a lock-in transition at about \( T = 1.9 \text{ K} \) [5]. Due to the hexagonal symmetry, there are three magnetic domains present.

In order to study the effect of magnetic frustration on the magnetic order in CePdAl in detail, we performed elastic neutron scattering measurements on single crystals. The experiments were conducted at the neutron source BER II at HZB. An observation of strong short-range order around and even far above the Néel temperature were indications of the presence of magnetic frustration. Moreover, we detected weak and broad peaks at “forbidden” magnetic reflections, where due to the magnetic structure factor no intensity should be present and which therefore might be a good measure for the degree of frustration. By applying magnetic fields within the basal plane, we investigated the stability of the magnetic order. The influence on magnetic domains and frustration in CePdAl will be discussed on this poster.

Magnetism in Eu(Fe$_{1-x}$Ir$_x$)$_2$As$_2$ Single Crystals: Complementary Neutron and X-Ray Studies

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Keywords: magnetic order, superconductivity, neutron diffraction, x-ray resonant magnetic scattering

Doped EuFe$_2$As$_2$ is a unique representative of the 122 type family of iron based superconductors due to the two magnetic sublattices and the strong coupling between spin-, lattice- and charge degrees of freedom. In present work, the magnetic ground states of the localized Eu$^{2+}$ spins in underdoped, nonsuperconducting Eu(Fe$_{0.94}$Ir$_{0.06}$)$_2$As$_2$ and optimally doped, superconducting Eu(Fe$_{0.88}$Ir$_{0.12}$)$_2$As$_2$ ($T_{SC} = 22$ K) were investigated by polarized and unpolarized single-crystal neutron diffraction measurements, respectively. The Eu$^{2+}$ spins in the underdoped ($x = 0.06$) crystal were found to order within the $ab$ plane in the A-type antiferromagnetic (AFM) structure. However, the Eu$^{2+}$ spins in the optimally doped ($x = 0.12$) crystal were found to be ferromagnetically aligned along the $c$ direction with an ordered moment of 7.0(1) $\mu_B$ at 2.5 K, coexisting with the bulk superconductivity. In addition, compared with the parent compound EuFe$_2$As$_2$, which shows the AFM order of the Fe$^{2+}$ moments below $T_{N,Fe} = 190$ K, $T_{N,Fe}$ was significantly suppressed to 85(2) K and completely suppressed, by 6% and 12% Ir doping, respectively. Most strikingly, the x-ray resonant magnetic scattering measurements on the underdoped ($x = 0.06$) crystal revealed the magnetic polarization of the $5d$ Ir dopant atoms and suggested a possible interplay between the localized Eu$^{2+}$ moments and the conduction $d$ electrons on the FeAs layers.

References:
[2] W. T. Jin, Y. Xiao, Y. Su, S. Nandi, W. H. Jiao, G. Nisbet, S. Demirdis, G. H. Cao, and Th.Brückel; Magnetic polarization of Ir in underdoped nonsuperconducting Eu(Fe$_{0.94}$Ir$_{0.06}$)$_2$As$_2$; Phys. Rev. B, 93, 024517 (2016); DOI: 10.1103/PhysRevB.93.024517
Magnetic Structure of Atomically Exchange Biased Dy\textsubscript{20}Co\textsubscript{80} Film

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Keywords: polarized neutron reflectometry, exchange bias, magnetism, thin films

Abstract: Amorphous Rare-Earth - 3d Transition Metal alloys (RE-3d TM) are fascinating magnetic materials due to the easy, straight-forward tunability of their magnetic properties, depending on temperature and concentration ratio between the RE and 3d TM components. If the RE-3d TM alloy is made up of heavier RE, both elements will be antiferromagnetically coupled by an atomic exchange interaction and show a temperature dependent resulting magnetization influenced by the different behavior of both sites. For Dy\textsubscript{x}Co\textsubscript{100-x}, it results into a transition from the Dy to a Co dominant magnetic phase with increasing temperature where the compensation temperature between both sites can be adjusted by the concentration ratio between both elements\textsuperscript{1,2}. A novel atomic exchange bias\textsuperscript{3} was found in a thin Dy\textsubscript{20}Co\textsubscript{80} film of about 50 nm resulting in a wing hysteresis loop with the origin of the loop shifted by 4 T. The analysis of detailed x-ray magnetic circular dichroism (XMCD) measurements traced back the effect to different surface and bulk magnetic configurations of surface and bulk. The occurrence of the wing shaped hysteresis loops shows that the surface and the bulk are strongly magnetically coupled by the atomic exchange interaction keeping the two magnetic states stable in a certain magnetic field range and suggest a non-collinear magnetic configuration in the intersection. The surface contribution dominates at higher fields and couples the magnetization of the bulk via the Zeeman interaction into the same direction, at lower fields, the bulk part dominates and the surface contribution is coupled via atomic exchange interaction with the bulk. For gaining precise qualitative and quantitative information about both competitive interactions, information about the magnetic structure of the film and its internal coupling behavior is required as it cannot be extracted from XMCD data. Polarized neutron reflectometry (PNR) is ideally suited to investigate the magnetic depth profile of the film. The polarization analysis of the reflected neutron beam enables one to detect the depth dependent orientation of the magnetization in Dy\textsubscript{20}Co\textsubscript{80} film, in particular to trace the expected non-collinear configuration in the region between surface and bulk. Moreover, an in-depth analysis of PNR spectra taken at different temperatures allows to gain further information about the switching process and thus to monitor the changes in the magnetization profile that leads to the intriguing exchange bias effect observed in the system.

Depth-resolved investigation of structural and magnetic correlations in a polarizing supermirror by GISANS with polarization analysis

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Keywords: magnetic nanostructures, polarized neutrons, GISANS: Grazing Incidence Small Angle Neutron Scattering, DWBA: Distorted Wave Born Approximation

Polarizing supermirrors are used for the polarization of cold and thermal neutron beams. Our aim was to determine, in such a system, the dependency of the lateral correlation length of the spin misalignment as a function of the layer and as a function of the applied magnetic field. For this purpose, we performed polarized GISANS with polarization analysis on this system using the MARIA instrument. The real advantage of MARIA is its unique ability to measure GISANS with polarization analysis up to large enough \(Q_z\) -values. An example of such a measurement is displayed in the Figure. In the non-spin flip channels, the signal stems mainly from scattering by interfacial roughness and, in the spin-flip channels, the signal stems from scattering by magnetic fluctuations. Simulation of the data within the DWBA obtained using the BornAgain software (http://www.bornagainproject.org) will also be presented.

Figure: Example of GISANS with polarization analysis. The four pictures correspond to different states of the neutron before and after interaction with the sample.
Spin-wave and Electromagnon Dispersions in Multiferroic MnWO$_4$ as Observed by Neutron Spectroscopy

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Keywords: spin-wave dispersion, electromagnon excitation, inelastic neutron scattering

So-called spin-driven ferroelectrics, for which the inversion symmetry is broken in the ferroelectric phase due to the appearance of a particular magnetically ordered state, provide a path to the required coupling$^{1,2}$. As a prototypical multiferroic material with spiral magnetic order, MnWO$_4$ has been widely studied concerning its magnetic and ferroelectric properties$^{3,4}$. However, a deeper insight into the coupling between the electric and magnetic degrees of freedom can be gained by studying not only the respective order but also the excitation spectra. In present work, high resolution inelastic neutron scattering reveals that the elementary magnetic excitations in multiferroic MnWO$_4$ consist of low energy dispersive electromagnons in addition to the well-known spin-wave excitations. The latter can well be modeled by a Heisenberg Hamiltonian with magnetic exchange coupling extending to the 12$^\text{th}$ nearest neighbor. They exhibit a spin wave gap of 0.61(1) meV. Two electromagnon branches appear at lower energies of 0.07(1) meV and 0.45(1) meV at the zone center. They reflect the dynamic magnetoelectric coupling and persist in both, the collinear magnetic and paraelectric AF1 phase, and the spin spiral ferroelectric AF2 phase. These excitations are associated with the Dzyaloshinskii-Moriya exchange interaction, which is significant due to the rather large spin-orbit coupling.

References:
[5] Y. Xiao et al., to be published.
Magnetic structures and magnetoelastic coupling of Fe-doped hexagonal manganites LuMn$_{1-x}$Fe$_x$O$_3$ ($0 \leq x \leq 0.3$)

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Keywords: neutron powder diffraction, multiferroics, structure

We have studied the crystal and magnetic structures of Fe-doped hexagonal manganites LuMn$_{1-x}$Fe$_x$O$_3$ ($x = 0, 0.1, 0.2,$ and $0.3$) by using bulk magnetization and neutron powder diffraction methods. The samples crystallize consistently in a hexagonal structure and maintain the space group $P6_3cm$ from 2 to 300 K. The Néel temperature $T_N$ increases continuously with increasing Fe-doping. In contrast to a single $\Gamma^4$ representation in LuMnO$_3$, the magnetic ground state of the Fe-doped samples can only be described with a spin configuration described by a mixture of $\Gamma^3$ ($P6_3'cm'$) and $\Gamma^4$ ($P63'c'm'$) representations, whose contributions have been quantitatively estimated. The ordered moment at base temperature amounts to about 3.2 to 3.5 $\mu_B$ per transition metal ion, nearly independent of the doping ratio. The drastic effect of Fe-doping is highlighted by composition-dependent spin reorientations. A phase diagram of the entire composition series is proposed based on the present results and those reported in literature. Our result demonstrates the importance of tailoring compositions in increasing magnetic transition temperatures of multiferroic systems.
The Effect of Rare Earth Substitution on the Structural and Magnetic Behavior of the Double Perovskite Oxides A$_2$FeCoO$_6$

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Keywords: double perovskites, structure, magnetism

The materials of the structure class called double perovskites have always fascinated the material researchers as they can accommodate a number of additions in their structure with varying valancy and ionic radii. The B-site ordering in them demands the optimization of charge (valancy) of the component cations and their ionic radii\textsuperscript{1}. The less ordered ones are also important as they can be utilized in many ways by tuning the stimuli (viz. electric field, magnetic field or heat energy). They are capable of exhibiting wide variety of properties like magnetoresistance, magnetocaloric effect, ferroelectric effect, superconductivity, etc. There have been a few reports on the double perovskites with B-site cations from the same row of the periodic table as there will be difficulty in achieving long range ordering. The double perovskites with Fe-Co in B-site belong to the strongly correlated systems with very peculiar physical properties as they are almost similar in ionic size. Also, as Fe and Co have similar structure factor for X-rays, it is almost impossible to see the B-site ordering with X-rays. The earlier reports on the well-studied Sr$_2$FeCoO$_6$ system reveals a spin glass phase near 75 K and magnetoresistance value of 63\% at 14 K in 12 T field. The presence of intermediate spin state of Co$^{3+}$ ion induces Jahn-Teller (JT) distortion in this system.\textsuperscript{2-4}. The motivation behind the current study is to elucidate the effect of rare earth substitution on the A-site of Sr$_2$FeCoO$_6$ on the magnetic properties and structure with temperature.

All the samples were prepared by citric acid based sol-gel method followed by annealing. The phase purity and crystallinity were studied from rietveld refined powder X-ray diffraction data and SEM-EDS analysis. The magnetic measurements were carried out in a commercial SQUID VSM. The rare earth substitution alters the magnetic as well as structural environment of the system drastically due to the change in A-site cationic size and charge. Compositions with higher ionic radii were found to be in rhombohedral $R3/c$ system at room temperature, while the others were formed in orthorhombic $Pnna/Pbnm$ space group. The unit cell volume is found to be decreasing as we go along left to right in the lanthanide series as expected. The exchange bias effect is observed in some of the samples, even at low cooling fields and is found to be increasing with the cooling field. None of the samples show magnetic saturation for higher fields up to 7T even at low temperatures up to 5K. At low fields (typically 100 – 1000Oe) they exhibit first order transitions in the temperature variation magnetization curves. Due to the complex magnetic behavior obtained with SQUID magnetization studies suggests neutron diffraction as the candidate to elucidate the underlying magnetism in the system. Also B-site ordering can be understood with the help of neutrons. The peculiar magnetic properties shown by the members of A$_2$FeCoO$_6$ nominate them for spintronic applications.

References
Neutron Diffraction to Distinguish Between Symmetry Lowering and Renninger Effect: An Example of Multiferroic Ba$_2$CoGe$_2$O$_7$

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Keywords: multiple diffraction, Renninger effect, Umweganregung, neutron diffraction, multiferroic melilite

Multiple diffraction in single crystals can generate reflections that are space-group forbidden by the Umweganregung process (the Renninger effect). Thus, there is always a risk of misinterpreting those reflections during crystal structure analysis. Compared to the X-ray case, neutron multiple diffraction has received relatively little attention in the literature. Here, we focus on this problem on example of the melilite Ba$_2$CoGe$_2$O$_7$. For a symmetry-consistent theoretical description of its multiferroic phase a precise knowledge of the crystal structure is a prerequisite. In a previous synchrotron X-ray diffraction experiment, forbidden reflections were found and interpreted by tetragonal-to-orthorhombic symmetry lowering of the compound$^1$. Here, we present the results of single-crystal neutron diffraction studies to differentiate between genuine symmetry lowering and multiple diffraction. The room-temperature experiments were performed with both hot (0.793 Å, HEiDi@MLZ) and cold (4.488 Å, MIRA@MLZ) neutrons. Multiple diffraction patterns (using so-called $\psi$-scans, Fig. 1) were simulated with the software UMPEG$^2$. It also allows to estimate some instrumental (e.g. beam divergences, wavelength spread) as well as sample-specific (e.g. mosaicity) parameters. Comparison of the experimental data with simulations shows that the scattered intensities detected at the positions of the forbidden reflections are entirely due to multiple diffraction$^3$. Thus, the crystal structure of Ba$_2$CoGe$_2$O$_7$ at room temperature is perfectly described by the tetragonal space group $P\overline{4}2_1m$ without any symmetry lowering.

Fig. 1. $\psi$-scans of the forbidden (100) reflection of Ba$_2$CoGe$_2$O$_7$ according to calculation (top) and the single-crystal neutron diffraction experiment (bottom) with a long wavelength $\lambda = 4.488$ Å

References:

Emergent Single Magnetic State in Mixed Valence Manganite Heterostructures

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Keywords: Polarized neutron reflectivity, Interface effect

Strongly correlated electron systems, like ferromagnetic La₁/₃Sr₂/₃MnO₃ (LSMO) and La₁/₃Ca₂/₃MnO₃ (LCMO) with considerably different magnetic ordering temperatures (Tₐ₉LSMO = 380 K [1] and Tₐ₉LCMO = 260 K [2]), are promising materials for novel spintronic devices. The mechanism for magnetization in both materials is based on the superexchange [3] and double exchange interaction [4], leading to a possible mutual influence at interfaces between LSMO and LCMO layers.

Performed polarized neutron reflectometry measurements on LSMO/LCMO thin film heterostructures reveal a complex magnetic behavior, like an induced magnetic ordering in the LCMO layer due to the interface to LSMO. High above the Curie temperature of LCMO, the LSMO layers behave as free magnetic layers but in the proximity to Tₐ₉LCMO these individual LSMO layers begin to couple. Finally, at the LCMO Curie temperature a single magnetic state emerges, which cannot be explained through electronic transport alone.

We will present the results of polarized neutron reflectometry measurements done on LSMO/LCMO multilayers.

References:

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Fe Layer Induced Ferromagnetism in Pd: An In-Situ Polarised Neutron Reflectometry Study

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Magnetic data storage systems are based on thin magnetic layers and heterostructures. For improving the functionality of existing devices and to develop new ones, a deep understanding of the properties of these layers and the coupling between them is essential. In this context not only the classic room temperature ferromagnets are of interest but also elements which can be polarised, like Pd, in which induced magnetism is observed when it is brought into contact with Fe. To monitor the structural and magnetic properties during the deposition process, in-situ polarised neutron reflectometry (PNR) is used as a novel analysis technique. These PNR experiments were carried out at the neutron reflectometer AMOR at PSI (Switzerland) using the Selene focusing optics to reduce measurement times.

In this contribution the evolution of the magnetism in polycrystalline Pd/Fe/Pd thin film systems during growth will be presented. For as little as a single monolayer of Fe deposited onto an initial Pd layer a high induced magnetic moment of approximately 1µB per unit cell was found in the Pd at the interface whereas the magnetic moment of the Fe is small compared to its bulk value. With more Pd deposited on top of the Fe layer, the magnetic moment of Fe increases while the induced magnetism in Pd decreases. An induced magnetisation of Pd was observed for a region of 7.5 Å on both sides of the Fe layer with a magnetic moment which decreases with increasing distance to the Fe/Pd interface. Additional TEM measurements show that the interfaces between Fe/Pd are sharp and it can be excluded that the magnetisation results from inter-diffusion or Fe clusters at the interface.
Polarized Specular Reflectivity and Off-Specular Scattering from Artificially Designed Magnetic Domain Patterns

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Keywords: magnetic pattern, domain-walls, polarized neutrons, off-specular scattering,

We report on polarized neutron specular reflectivity and off-specular scattering from artificially designed lateral magnetic domain patterns. Tailored magnetic domains on micrometer scales and emerging stray field landscapes can function as platforms for various lab-on-a-chip applications. He-ion bombardment of photo-lithographically patterned, exchange biased magnetic thin films in external magnetic fields leads to micrometer sized domains with chosen width and structurally homogeneous surfaces. Artificial stray field landscapes (Figure 1a) are created by charged domain walls between in-plane magnetic domains with head-to-head and tail-to-tail configuration.1,2 We will present polarized neutron reflectometry and off-specular scattering as a tool to investigate the global magnetic state of the sample in different magnetic configurations. Specular reflectivity reveals the laterally averaged depth dependence of structure and magnetism in the thin-film system. Off-specular scattering (Figure 1b) obtained in different magnetic configurations probes the lateral distribution of magnetic moments. Unlike local probes, like Kerr-effect microscopy or magnetic force microscopy, the complete surface area is sampled in a single measurement, revealing domain contrast, correlation lengths, magnetization directions and disordered moments.

In addition to the scattering of the domains, we report on resonant spin-flipping processes of the evanescent wave in the samples. In a narrow region just below the critical edge of total reflection, enhanced spin-flipping and off-specular scattering is observed with a location corresponding to the length scale of the formed quantum-well.

References:

![Figure 1: a) Schematic magnetic domain configuration. b) Spin-flip specular and off-specular scattering of domains with 2μm width.](image)
Magnetic Structure of Iron Oxide Nanoparticle Assemblies

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Keywords: polarized neutron reflectometry, small-angle scattering, magnetic nanoparticles

Magnetic nanostructures are fundamentally interesting concerning the understanding of dipolar coupling, exchange coupling, as well as for technological applications in information technology and spintronics. Thin film engineering with targeted magnetic properties on the nanometer scale is particularly attractive for the realization of magnetic sensors and high density recording media 1, 2. Depending on the deposition technique and the individual nanoparticle properties, homogeneously ordered and disordered nanostructures are prepared from a large variety of materials 3. Spin-coating of nanoparticle dispersions, for example, yields extremely homogenous particle layers across several square centimeters, having a certain degree of order induced by the substrate 4.

In this contribution, we will present a combined small-angle scattering (SAS) and polarized neutron reflectometry (PNR) study of iron oxide nanoparticles and their assemblies. Polarized SANS (SANS-POL) by magnetic nanoparticle dispersions yields information on the magnetic properties of the individual particles 5. PNR gives access to the magnetic depth profile of a closely packed nanoparticle assembly and possible interaction effects 6. The combination of both techniques gives insight into the impact of nanoparticle interactions on their magnetic properties. Our results are confirmed macroscopically by vibrating sample magnetization measurements.

References:
Neutron Scattering Investigation of Rare Earth Pyrochlore Iridates and Hafnates

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Keywords: neutron powder diffraction, pyrochlore, frustration

The rare-earth pyrochlore oxides have been intensively studied over the past few decades due to geometrical frustration. Pyrochlore iridates and hafnates A2B2O7 (A= rare earth ion, B=Ir, Hf), in which both the A site and the B site consist of corner-shared tetrahedra, are of particular interest due to the presence of both strong spin-orbital coupling and geometrical frustration.1 A2Ir2O7 displays an intriguing metal-to-insulator transition (MIT) driven by both thermodynamics and internal chemical pressure, which is suggested to coincide with possible magnetic order at the Ir4+ site.2 Moreover, the two sublattices of A3+ and Ir4+ might be magnetically coupled thus leading to novel magnetic behaviors. As a counterpart, A2Hf2O7 compounds are insulator and the Hf4+ ion is nonmagnetic. In the present work, pyrochlore iridates and hafnates within light rare earth Pr and Nd were synthesized by solid states reaction. Combined X-ray and neutron powder diffraction refinement suggest the samples are stoichiometry. Their magnetic ground states were studied by Polarized neutron scattering on DNS. ‘All-in-all-out’ and ‘two-in-two-out’ magnetic configurations were observed in different sample at low temperature.

Magnetism at Heinz Maier-Leibnitz Zentrum (MLZ)

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Keywords: neutron, instrumentation, structure, dynamics

Neutron scattering is the leading technique for measuring magnetic correlations in solids. It can be used as a direct probe for studying magnetic order and magnetic moment coupling in frustrated or low dimensional materials. It can also prove dynamic correlations, their lifetime and diffuse scattering in the absence of magnetic order. The suite of neutron instruments at the MLZ, located at the research reactor FRM II in Garching, provides a unique opportunity to study such magnetic phenomena. The MLZ provides free beam time for scientific use at its instruments for everybody (according to the decision of the review panel and under the condition of publishing the results).

Our recent highlights cover broad variety of scientific fields like frustrated magnetism (e.g. bilayer pervoskite Sr$_x$Fe$_2$O$_7$ [1] or lifetime of spin waves in 2D and 3D systems [2]), magnetic excitations in insulators (e.g. in cobalt oxides [3]), quantum phase transitions (e.g. absolute zero vibrations in superfluid helium [4] or Higgs mechanism in quantum spin ice [5]), superconductivity (e.g. nematic correlations in high temperature superconductors [6]) or novel magnetic states (e.g. helimagnons, skyrmions [7,8]).

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References:

Motion in Diffusivity Landscapes: Towards Analytic Correlation Functions

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Keywords: fit functions, anomalous diffusion, heterogeneous media

Anisotropic and anomalous diffusion in heterogeneous environments – governed by a landscape of energy and diffusivities – is a relevant and frequently studied phenomenon in experiment, simulation and theory. A paradigmatic example is water diffusion through hydration shells around biomacromolecules or membranes. Analytical fit functions for complex systems such as these are rare, although they would be interesting to connect conceptual ideas to experimental results.

We report on an approach to recover effects of geometry and position-dependent diffusivity by approximate solutions well-suited for data fitting.¹ In particular, we map the motion through a complex landscape on the stochastic switching in a network of dynamical states with approximated internal dynamics. First, we derive an analytical function for an isotropic motion switching in time between different diffusive states, which has been successfully used to describe the hierarchical subnanosecond dynamics of proteins.² Second, we extend the approach towards anisotropic systems such as layer structures, aiming for e.g. a description of anisotropic water diffusion around membranes.³ Finally, we discuss the challenges, limitations and potentials of the outlined method for data analysis integrating experimental and theoretical knowledge.

Figure 1: Mapping motion in complex landscapes onto a network of dynamical states, correlation functions can be determined. Graphics taken from [1].

Neutron Reflectometry Investigation of Adsorption Behavior in a Polyelectrolyte/Surfactant mixture and its Correlation to Foam Film Stability

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Keywords: neutron reflectometry, foams, adsorption, surfactants, polyelectrolytes

The properties of foams are of interest for many industrial applications such as enhanced oil recovery and in personal care products and are therefore the subject of many studies. A way to produce stable foam films is to mix surfactants with oppositely charged polyelectrolytes, as highly surface-active complexes can be formed with the two compounds.

Extensive research on such mixtures was already performed with the focus on very flexible polyelectrolytes, as PAMPS¹². However, it is still unclear what the influence of the backbone rigidity of the polyelectrolyte on the resulting foam film properties is. In this work a mixture of a newly synthesized polyelectrolyte (sPSO₂-S220)³ with a stiffer backbone is used and mixed with the cationic surfactant C₁₄TAB.

The system has been investigated by means of neutron reflectometry experiments to get insight into the exact composition of the adsorbed sPSO₂-S220/ C₁₄TAB complexes at the air/water interface. These results were correlated to foam film stabilities of the respective mixtures ⁴.

Those measurements have shown a stronger adsorption and thus a higher synergistic effect for the sPSO₂-S220/C₁₄TAB mixture then for mixtures with more flexible polyelectrolytes. This is also reflected in significantly more stable foam films. Neutron reflectometry experiments showed the formation of 1:1 complexes at the surface.

References:
Shear-induced transformation of polymer-rich lamellar phases to micron sized vesicles investigated by small angle scattering

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Keywords: small angle scattering, light, neutrons, X-rays, viscosity, rheology, lamellar phase

The influence of adding an amphiphilic triblock copolymer to a cationic microemulsion results on the one hand in an increasing viscosity and on the other hand in complexer phase behavior with an enhanced lamellar phase. This is a common feature of amphiphilic block copolymers related to the efficiency boosting effect[1].

The aim of this work was to fabricate multi-lamellar-vesicles (MLV) out of a triblockcopolymer-rich lamellar phase applying stress and their identification via small angle scattering. Such large vesicles are of potential interest for the usage as drug delivery systems. The four-component system which has been used in this work consists of A) a polar component D2O, B) an apolar component o-xylene, C) an amphiphilic triblock-copolymer PE9400 (EO21-PO47-EO21) as the surfactant and D) a cationic co-surfactant namely C8TAB (n-octyl-trimethylammonium bromide). The polymer-to-oil ratio and the co-surfactant-to-polymer ratio were varied systematically to find the regions where the sample exhibits a lamellar phase. By increasing the polymer content a huge lamellar region arises, which can easily be identified by small angle scattering (SAS, Figure 1) or by its typical birefringence effect. It has been found that shearing a lamellar sample with shear rates between \( \gamma = 1 – 100 \text{ s}^{-1} \) results in the formation of spherical structures with radii between 2 and 10 µm. The size of the fabricated vesicles is inversely dependent of the applied shear rate and of the amount of co-surfactant. The change in size was detected directly via SAS and microscopy, and indirectly via the increase of viscosity under the applied shear stress. Due to their large size, it is difficult to investigate such big structures via SAS. However light scattering (SALS, Figure 2) and microscopy techniques (phase-contrast, difference interference contrast) are perfectly suited for the investigation of such large vesicles. The change of the lamellar structure under shear stress can be investigated by rheo-small angle neutron scattering (rheo-SANS, Figure 1).

References:
Simultaneous SANS and FTIR Measurements on Host-Guest Polymeric Frameworks

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Keywords: FT-IR, SANS, co-crystals, syndiotactic polystyrene.

A novel method for the simultaneous measurement of small angle-neutron scattering and Fourier transform infrared spectroscopy (SANS/FTIR) has been developed at the KWS-2 SANS diffractometer of JCNS. This combination of techniques is particularly desirable for the study of structural changes occurring in soft-matter and biological systems due to the variation of composition or external fields, when two or more parameters or features can be simultaneously investigated in such complex systems. Additionally, it helps for an accurate characterization of samples by SANS considering that the sample quality is monitored by FTIR and an undesired sample change or degradation due to small variations of external factors (such as temperature, pressure, etc.) will be known and taken into account for either a proper data analysis or a repetition of the investigation using fresh sample. Phase transition phenomena, studies of morphology and conformation of molecules in confinement or under special external conditions (irradiation, temperature, pressure) are the fields which will benefit at KWS-2 from this new experimental approach.

The feasibility test of the method was carried out through a study of simultaneous time-resolved in-situ SANS and FTIR investigations of syndiotactic-polystyrene (sPS) co-crystals with small guest molecules¹⁻³. The combined approach helped for a better understanding of the structural changes occurring by heating of samples (sPS films doped with short PEGDME molecules with a molecular weight ranging between 178 g/mol and 500 g/mol) and, above all, to determine distribution and conformation of the guests inside the amorphous and crystalline regions of the host polymeric matrix and their variation with temperature.

In this contribution, we will present the concept and technical details of the combined experimental approach, supported by results obtained during the commissioning at KWS-2. Furthermore, we will report the achieved improvements from the simultaneous combined FTIR/SANS in a performed experimental test after installing: 1. a new fixed support and sample holder to measure materials in different physical states; 2. a new optical system updated with the mounting of new parabolic mirrors (in order to optimize the beam divergence); 3. new windows, of different materials with a larger transmission wavelength range, a smaller neutron activation (so an increased degree of safety), a better thermal and chemical resistance and improved mechanical strength compared to the KBr windows used so far.

Figure 1 - On the left side, experimental set-up for simultaneous FTIR/SANS measurements. The sample is irradiated collinearly with a neutron and an infrared beam. On the right side, FT-IR instrument with new updated optical system.

References:
[2]. F. Kaneko et al., Chemistry Letters, 44, 497 (2015); http://doi.org/10.1246/cl.141179
Refinement of Molecular Dynamics Simulations with Scattering Data

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Molecular Dynamics simulations are based on so-called force fields in which the interaction between different atoms is parametrised. There are two distinct parts to a force field: (1) the functional form of the interactions (e.g. the choice to use a Lennard-Jones potential to account for van der Waals interactions) and (2) the parameters of these functions (e.g. the coefficients of the $r^6$ and $r^{12}$ term). Normally, this parametrisation is based on macroscopic quantities like the heat of evaporation – but with scattering techniques, it is possible to measure the microscopic properties of molecules directly which should make a much more direct and reliable parametrisation of the force fields possible.

The idea to refine computer simulations with scattering data is the basis of several well-established techniques that strive to refine the structure of materials. However, they either completely abolish the concept of interaction potentials (Reverse Monte Carlo) or alter the functional form of the interactions in a fairly uncontrolled way (Empirical Potential Structure Refinement). These methods have the advantage that they are very computationally efficient when taking the scattering data into account but the drawback that the result is a non-unique structure which is compatible with the data but doesn't have a physical justification within the model.

In this project, it is explored if it is feasible to keep the functional forms of the potentials and only change the parameters of these functions. Also the focus of the optimisation is slightly shifted: instead of fitting the structure, it is the pico- to nanosecond dynamics using incoherent quasielastic neutron scattering measurements of water taken at the time-of-flight spectrometer TOFTOF at the MLZ.
Neutron Reflectivity Investigation on Thermo-responsive Copolymers Embedded with Magnetic Nanoparticles

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Keywords: neutron reflectivity, block copolymer, thermos-responsive, magnetic nanoparticles

Nanostructured hybrid composites consisting of copolymer template with embedded nanoparticles are promising materials in many fields due to their novel chemical and physical properties. For the purpose of magnetic applications, magnetic nanoparticles are favored due to their lower magnetic remanence and coercivity, compared with bulk magnetic materials. The realization of desired magnetic property has an indispensable dependence on the arrangement of nanoparticles and can be tuned by employing diblock copolymer as host matrixes. Various ordered nanostructured templates can be achieved from the phase separation of diblock copolymer composed of two chemically incompatible blocks. Being coated with one particular type of polymer chains, magnetic nanoparticles can have selective affinity to one of the blocks and get well distributed within this domain. So far, thermal-responsive copolymer templates are rarely investigated in spite of the high potential applications. Recently, we investigated the nanostructure and magnetic behavior of hybrid dry films consisting of magnetite \((\gamma-\text{Fe}_2\text{O}_3)\) nanoparticles coated with polystyrene chains and an asymmetric diblock copolymer polystyrene\(\text{61}-\text{block-\text{polyN-isopropylacrylamide}}_{115}\) (PS-b-PNIPAM). Results showed selective incorporation of nanoparticles into PS domains and morphological evolution from parallel cylinder to perpendicular one at low concentrations. And a superparamagnetic behavior was found irrespective of nanoparticle concentration. On the basis of understanding the morphology of these films, we want to extend our investigation from dry films to wet ones, in order to explore the swelling behavior of films and its impact on the magnetic property. In the planned experiment, neutron reflectivity and grazing incidence small angle neutron scattering will be used to detect the morphology of films as a function of temperature in a humid atmosphere. In addition, polarized neutron will also be employed to probe the magnetic structure in this investigation.

[1] Yuan Yao, Ezzeldin Metwalli, Martin A. Niedermeier, Matthias Opel, Chen Lin, Jing Ning, Jan Perlich, Stephan V. Roth, and Peter Müller-Buschbaum; Nano- and Microstructures of Magnetic Field-Guided Maghemite Nanoparticles in Diblock Copolymer Films; \textit{ACS Appl. Mater. Interfaces}, 6, 5244 (2014); DOI: 10.1021/am500597t


Effect of alcohol treatment on the morphology and performance of organic solar cells

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Keywords: energy, solar cell, structure, GISANS

Organic solar cells are an interesting alternative to conventional silicon based solar cells as the feature new possibilities introduced by using a different class of materials namely polymers. Instead of expensive ultra-high vacuum technologies, fabrication can be done at room temperature, using wet chemical processing, and thereby enabling usage of methods such as roll-to-roll printing. As a consequence, the production of organic solar cells has the potential to become very cheap and easy. Moreover, the use of polymers allows for flexible solar cells and light weight devices, which will be usable in a very different fashion as compared to the immobile silicon solar panels. In addition, the energy payback times of organic solar cells are significantly shorter as compared to the today’s silicon solar cells. However, despite all these significant advantages of organic solar cells, still fundamental knowledge is very limited. In particular, it is challenging to detect the complex morphologies, which are necessary to have high efficiency organic solar cells. The combination of grazing incidence small and wide angle scattering (GISAS and GIWAS) allows for overcoming these challenges.

The environmentally friendly alcohol treatment of organic solar cells using a low bandgap copolymer called PTB7 and the fullerene derivate PC$_{71}$BM is studied. Different alcohols are tested and besides the most commonly used methanol treatment, other alcohols such as ethanol, 2-propanol, and 1-butanol also improve the device performance as compared to untreated solar cells. Changes of the surface structure caused by the alcohol treatment are probed with AFM and the modification of inner film morphology is probed by time of flight-grazing incidence small angle neutron scattering (TOF-GISANS). UV/Vis measurements show that the thickness of all films remains unchanged by the different solvent treatments. Thus, the enhanced device performance induced by the alcohol treatments is correlated to the reconstruction of the inner film structures probed with TOF-GISANS and the modified energy levels at the interfaces between the two components of the active layer and the aluminum electrodes, evident by the enhanced short-circuit current and open-circuit voltage of the I-V curves.

References:
Aqueous Solutions of Poly(ethylene oxide) - Crossover From Ordinary To Tricritical Behavior

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Keywords: SANS, aqueous polymer solutions, phase behavior

Aqueous solutions of 0.45 to 280 kDa poly(ethylene oxide) (PEO) near their critical concentrations were studied with SANS in a temperature interval from 11.8 to 95.7°C. This kind of studies is relevant as “the behavior of PEO in aqueous environment is the key to its importance in biomedical and biotechnological applications” as has been phrased by J.M. Harris in ref [1].

Thermal concentration fluctuations of PEO delivered susceptibility and correlation length which allowed identifying the areas of mean field, 3D-Ising, and tricritical Θ-point behavior in dependence of the PEO molar mass and concentration. The predicted scaling laws of the critical amplitudes of susceptibility and correlation length for semi-dilute good solution could be confirmed in the areas of 3D-Ising and tricritical Θ-point behavior whereas the chain conformation was different. In tricritical case chain conformation behaves as expected whereas in 3D-Ising case the chains are stronger compacted than a Gaussian coil. At the Θ temperature all chains show near ideal conformation. A small amount of PEO of the order of 10⁻⁵ is forming compact PEO aggregates.

The figure shows the critical temperature – critical concentration plane of the phase diagram of the aqueous (D₂O) PEO solution. The critical point (⊙) at Φ \( \rightarrow 0 \) represents a tricritical point as it is the end point of two lines of critical points. (i) A line of lower critical solution temperatures (LCST) of a continuous first-order phase transition (●) and negative second virial coefficient (A₂ < 0). (ii) The λ-line depicted as red arrow at Φ \( \rightarrow 0 \) (thereby zero order parameter) and A₂ > 0 represents the critical point of a second-order transition. The corresponding green line represents the Θ-temperature (◆) for the solutions as derived from the parameters T_C and N.

References:

Polymer dynamics within soft confinement

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Keywords: Neutron spin echo, microemulsion, polyethylene glycol, confinement

Microemulsions in the droplet phase are a well suited system to provide a three dimensional soft confining geometry on the length scale of several Nano metres. Due to the softness of the confinement molecules which are introduced into the microemulsion droplets will change structural and dynamical properties of the confinement. In case of water in oil microemulsions stabilised by the an-ionic surfactant AOT we studied these changes for the case of the water soluble polymer Polyethylene oxide (PEO) enclosed in droplets in a range of droplets sizes.[1,2,3]

Having precisely characterised the confinement we now move on and present our findings on the structure and dynamics of the confined polymer PEO, investigated by small angle neutron scattering (SANS) and neutron spin echo spectroscopy (NSE). The SANS results show a significant increase of the polymer’s radius of gyration, which is related to an attractive interaction between PEO and the AOT head groups. Furthermore the internal correlations of the polymer change, since the scattering data can no longer be described by a Debye-Function implying a Gaussian conformation of the polymer. Instead a model for collapsed polymer chains has to be used resulting in a reduction of the Flory parameter to 0.33 corresponding to the bad solvent limit. The actual size of the confinement does not seem to be important for the polymer properties instead the concentration seems to dominate the structure.

For the NSE data the effective diffusion coefficient was investigated. For small scattering vectors q thus \( q \cdot r_c \leq \pi \), with \( r_c \) the radius of the microemulsion droplets, pure droplet diffusion is observed. At larger q values the dynamics show a significant increased effective diffusion coefficient due to the polymer dynamics.

Unexpected Swelling of Polyelectrolyte Brush/Multilayer Composites in Humid Conditions

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Keywords: Polyelectrolyte multilayers, polymer brushes, ellipsometry, neutron reflectometry, humidity

Introduction: Polymer coatings open a wide range of smart and active surfaces. Among possible preparation methods, the Layer-by-layer technique is an easy and versatile tool to functionalize a charged surface with a polyelectrolyte multilayer (PEM) with a thickness ranging from few nanometers up to micrometers¹. The large variety of polyelectrolytes opens different fields of applications for PEMs, for example antireflective coatings, antifouling coatings or carrier systems for specific target molecules.

Besides multilayers, polymer brushes can be also applied for surface functionalization. In contrast to physisorbed PEMs, brushes are covalently bound the surface. In particular, the grafting-from approach opens the opportunity to prepare well defined end-grafted polymer brushes with high grafting density and low polydispersity². From this, robust coatings with specific functionality can be prepared, which can be applied as coatings for implants, nanostructured surfaces or surfaces with specific hydrophobicity. The combination of both, multilayers and brushes, leads to a robust and multifunctional coating³. The study of the response to external stimuli is of crucial importance for many technical applications.

Presented work: Here, we present the swelling behavior of a composite consisting of a PEM-coated brush in humid conditions. PEM and brushes show a different swelling behavior during increasing relative humidity (RH). Ellipsometry data show that the swelling degree for PEM-coated brushes is between the two bare systems. A defined contrast between both compartments of the composite cannot be investigated by ellipsometry due to the very similar refractive index. Therefore, we used neutron reflectometry to investigate PMETAC brushes coated with dPSS/PDADMAC PEM of different layer numbers. The study demonstrates that PEM diffuses into the brush during the layer deposition and diffuses inside the brush by increasing the RH. At high RH the PEM diffuses towards the film/air interface, while the brush beneath the PEM absorbs reversibly more water than the PEM on top, which is counterintuitive. Possible explanations will be given.

References:
Interactions of Polyelectrolytes with Oppositely Charged Microemulsion Droplets – the Effect of the Bridging Polyelectrolyte

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Keywords: interactions, structure, neutron scattering

By mixing polyelectrolytes (PE) with oppositely charged colloids one gains control of the structural and rheological properties of the system. In most applications, micelles are used as carriers for drug molecules, pollutants, reactants and similar substances that otherwise couldn’t be dispersed in the solvent. It is therefore of great importance to investigate the behavior of ‘loaded carriers’ as it can be different to that of normal (empty) micelles.

For this reason we studied the interactions of positively charged O/W microemulsion (ME) droplets that already contain a substantial load of solubilized molecules with negatively charged polyelectrolytes. With small-angle neutron scattering (SANS) it was possible to characterize the formed complexes in terms of size, shape and composition. Many different structures were observed especially in the polyelectrolyte excess part, which mainly depend on the mixing ratio and the chain length of the polyelectrolyte. We studied the effect of the polymer stiffness by employing Na-PAA as a flexible polyelectrolyte with a high charge density as well as stiff biopolyelectrolytes, such as carboxymethylcellulose (CMC). Further, the coupling between the persistence length of the PE and the curvature of the microemulsion droplet was examined in detail.

These experiments together give a comprehensive structural picture of the formed complexes and allow a detailed understanding of the interactions between charged ME droplets and polyelectrolytes. The ME – PE aggregates serve as a model system for complexes with a high solubilization capacity that find potential applications in formulations which contain a large amount of oil or hydrophobic agents with tailor-made viscosity and internal mobility, both required for optimized delivery.

Figure 1: Neutron scattering curves I(q) and corresponding structures of microemulsion – polyelectrolyte complexes for three different charge ratios $z = [-]/([+] + [-])$.

$\text{Figure 1: Neutron scattering curves } I(q) \text{ and corresponding structures of microemulsion – polyelectrolyte complexes for three different charge ratios } z = [-]/([+] + [-])$. 
Ionic Self-assembly of Polysaccharides and Alkylethoxycarboxylates: Complex Responsive Materials from Simple Building Blocks

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Keywords: small-angle scattering, complex materials, self-assembly, soft matter

Chitosan is a biofriendly positively charged biopolymer; Alkyl ethoxy carboxylic acids (C\textsubscript{i}E\textsubscript{j}CH\textsubscript{2}COOH) are an interesting class of surfactants, carrying the properties of ionic and nonionic surfactants. The components are responsive to pH and temperature, affecting their degree of ionization and the hydration of the EO shell. Moreover, size and shape of the self-assembled surfactant aggregates can be controlled by the lengths of the hydrophilic and hydrophobic blocks.

Chitosan and alkyl ethoxy carboxylic acids co-assemble into a surprisingly large variety of structures, depending on the mixing ratio, degree of ionization of the components, and spontaneous curvature of the surfactant aggregates\textsuperscript{1,3} (see Fig. 1). Particularly strong is the response of the system towards pH, with drastic changes in the physico-chemical properties of the system within less than one unit of pH. The observed richness in complex structures then not only allows to design smart materials that react sensitively to changes of external parameters like pH but also are in general interesting formulation systems, where the properties can be varied largely by the molecular composition and due to their biofriendliness are promising candidates for advanced formulations\textsuperscript{4}.

Fig. 1: Schematic representation of self-assembled structures formed in chitosan / alkyl oligoethylene-oxide carboxylic acids mixtures.

References:
Proton dynamics of phosphoric acid in the catalytic layer for HT-PEFC

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Keywords: neutron, dynamics, QENS

High-temperature polymer electrolyte fuel cells (HT-PEFC) are promising electrochemical energy converters. Because of the high operation temperature of 160° - 180°C HT-PEFCs have a high CO tolerance. [1]. Recent works on the development of HT-PEFC are focused mostly on the performance and technical optimization. However, the fundamental investigation of the proton conductivity will help to optimize performance and increase sufficiency of the fuel cells. For example, the understanding of proton diffusion mechanism in membrane electrode assembly (MEA) plays a key role in proton conductivity of fuel cells.

The present study focuses on dynamical properties of phosphoric acid (PA) in the catalytic layer for HT-PEFC. The catalytic layer is a composite material containing nanoporous carbon, poly(tetrafluoroethylene) (PTFE) and platinum (Pt) nanoparticles. Since the catalyst layer is in direct contact with the PBI membrane doped with PA it also contains some amount of acid, which is needed to provide good proton conductivity in the electrode. The contact between electrodes and the membrane is one of the important parameters, which influence the cell performance [1,2]. Knowledge about local PA dynamics and understanding the distribution of PA between structural elements of the catalyst could help to design more efficient electrodes for fuel cells.

Due to adsorption of the PA on the Pt particles the diffusion of protons in the catalytic layer can be different with respect to the membrane and bulk acid and thus, should be taken into account [3]. Using quasielastic neutron scattering (QENS), proton dynamics can be studied on local length scales from few Angstrom and up to 10 nm. Backscattering spectroscopy was used to directly probe the dynamics of protons in nanoseconds range. The three approaches are considered for description of proton dynamics: the random jump diffusion model, distribution of jump lengths and, finally, the trap model. Extracted parameters such as diffusion coefficient, activation energies and time constants gives insight about dynamical behaviour of the PA and its distribution in the catalytic layer.

References:
de Gennes Narrowing and Hard-Sphere Approach

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Keywords: neutron, MD simulations, dynamics, diffuson

The energy width $\Delta \omega$ of the quasielastic coherent dynamic structure factor $S(Q, \omega)$ for a simple liquid exhibits the oscillating dependence on wave number $Q$ with the sharp minimum at $Q_{\text{max}}$, known as de Gennes narrowing, corresponding to the maximum of the structure factor $S(Q)$. The only known expression for $\Delta \omega(Q)$ was derived for a dense hard-sphere (HS) fluid:\n
$$\Delta \omega_{\text{HS}} \approx \frac{D_E Q^2}{S(Q) \left[ 1 - j_0(Q \sigma_{\text{HS}}) + 2 j_2(Q \sigma_{\text{HS}}) \right]}$$  \hspace{1cm} (1)$$

Here $D_E$ is the Enskog’s diffusion coefficient, $\sigma_{\text{HS}}$ is the HS diameter, $j_0$ and $j_2$ are the first two even order Bessel spherical functions. Though this expression has been frequently used for the analysis of the experimental data obtained for liquid metals, until now, it has never been tested against a true HS fluid. A test performed by means of HS molecular dynamic simulations reveals significant discrepancy between the simulations results and the examined model. The main output of the analysis is the finding that the $\Delta \omega_{\text{HS}}(Q)$ behavior is defined in terms of the average cage size $<L_c>$, rather than of $\sigma_{\text{HS}}$. An empirical improvement of model (1) is proposed, which describes $\Delta \omega_{\text{HS}}(Q)$ with good accuracy in the $Q$-range corresponding to the first maximum of $S(Q)$ and for the packing fractions $\varphi = \frac{\pi n \sigma_{\text{HS}}^3}{6} = 0.43 - 0.49$.

One cannot expect that the HS approach can be applied to real liquids without any limitations. In order to explore these limitations, as the first step, the simulated $\Delta \omega_{\text{HSF}}(Q)$ has been compared with the results for the soft-spherical potential. The microscopic dynamics of the simulated soft sphere fluid shows significant difference in comparison to the HS system. Nevertheless, the diffusive mobility of soft spheres can be characterized within the HS approximation using an effective diameter, $\sigma_{\text{eff}}$, and this parameter can be found from $\Delta \omega(Q)$ at $Q \approx Q_{\text{max}}$. A similar result has been obtained for the neutron scattering data measured for liquid Al, Cs, Ni and Rb.

References:
Investigation of the Osmotic Deswelling of Polystyrene Microgels
by Polymer Addition

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Abstract: The addition of free polymer to a glassy colloidal dispersion leads to the creation of a fluid pocket in the phase diagram at high volume fractions between two glassy states - the "repulsive" and the "attractive" glass. This re-entry effect is extraordinarily strong for polystyrene (PS) microgel colloids in good solvent to which linear PS chains are added - a phenomenon which is still not understood [1]. As the re-entry effect is very robust and can be transferred to technical (aqueous) dispersions, a very interesting application for economic and ecological reasons [2], it is important to understand the physical origin of the differences between typical hard spheres, like PMMA particles [3], and the microgel system which has so far not been achieved. As the microgel particles are swollen in a good solvent typically by a factor 5-6 in volume, osmotic deswelling has been discussed as an obvious explanation, since this would lead to smaller effective volume fractions and resolve the apparent discrepancy with respect to the PMMA system. Measurements of a dilute deutero PS tracer in a highly concentrated protonated PS host via small angle neutron scattering (SANS), however, showed no difference in the formfactor when we increased the polymer concentration. The next obvious step is the examination of size and structure of the free polymer in the system and the structural changes it causes in the packing of the colloidal particles. The major part of the huge re-entry region of microgel colloids in mixtures with free polymer is located at effective polymer concentrations $c_{P,\text{free}}$ above the overlap concentration. This suggests that the concept of the radius of gyration $R_g$ becomes obsolete in semi-dilute polymer solutions and should be replaced by the screening length $\xi$. Therefore we determined with SANS the screening length of fully deuterated PS polymer in a contrast-matched host of microgel particles at a colloid volume fraction of $\sim$0.74. We find that the screening length of the polymer confined by the microgel matrix is much smaller than in pure polymer solution at identical reduced polymer concentration $c/c^*$. 

References:
Coarse-grained molecular dynamics and neutron reflectivity characterization of supported lipid membranes

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Keywords: neutron reflectivity, molecular dynamics, membranes, biophysics

Supported lipid bilayers on planar surfaces constitute an archetypical experimental system for the study of biological membranes. The widespread use of these ordered molecular layers (as evidenced in literature), is on one hand related to the simplicity of their preparation using the methods of vesicle fusion or Langmuir-Blodgett deposition and on the other hand to their compatibility with a multitude of surface sensitive experimental probes (including AFM, ellipsometry, quartz-crystal microbalance, surface plasmons and fluorescence methods). Neutron reflectivity at a solid/liquid interface has proven as an important experimental method for the investigation of such systems with the ability to provide sub-nanometer structural information perpendicular to the supporting plane. Traditionally reflectivity data are compared to theoretical curves of simplified models consisting of stratified layers representing the hydrophilic (lipid heads) and hydrophobic (lipid tails) parts of the bilayer. In the present work we explore the combined use of molecular simulations and neutron reflectivity for the characterisation of supported membranes. By performing coarse-grained molecular dynamics simulations based on the MARTINI force field of supported 1,2-dihexadecanoyl-sn-glycero-3-phosphocholine (DPPC) bilayers close to a hydrophilic substrate, we compared the obtained reflectivity profiles with neutron reflectivity data for this system at a series of temperatures above and below the main phase transition. The observed quantitative agreement between simulation and experiment after fine tuning the system’s interactions, for both the liquid and gel state of the lipids, showcases that the presented methodology may serve as a basis for the detailed and assumption-free investigation of more elaborate systems like membrane/nanoparticle or membrane/oligopeptide complexes.
Interaction of escin, cholesterol and ibuprofen with unilamellar DMPC vesicles as seen by small angle scattering.

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Keywords: small-angle neutron scattering, phospholipids, vesicles, escin

Phospholipids are the main component of biological membranes. The interaction of drugs, membrane bound proteins, and receptors are of major interest in industrial and pharmaceutical research. The function of membrane proteins depends on the lipid bilayers thickness ($d_l$) and its properties. We study the properties of the lipid bilayer in form of model membranes as unilamellar vesicles (ULVs) consisting of the phospholipid 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC). DMPC undergoes a phase transition at $T_m=23.6 \, ^\circ\text{C}$ [1] from a rigid gel to a 2D flexible fluid structure. The properties of the lipid membranes can be affected by the type of the inserted molecule into the membrane, as for example cholesterol or the non steroidal anti-inflammatory drug ibuprofen. Saponins are a divers class of natural, plant derived amphiphilic molecules with a peculiar molecular structure made of a hydrophobic scaffold and hydrophilic oligosaccharide chains. The incorporation of saponins reduces the cholesterol bioaccessibility by displacement of cholesterol molecules from bile salt micelles. We studied the pure saponin escin to demonstrate the effect of saponin incorporation on the lipid membrane in gel and fluid phase. The study is complemented with combinations of escin, cholesterol and ibuprofen. With small-angle neutron studies at the PA20 at LLB and small- and wide-angle X-ray scattering on an inhouse instrument we probe the vesicle main size parameters, such as vesicle radius, bilayer thickness, and phospholipid head group distance. The incorporation of escin induces similar to ibuprofen [2] bilayer thinning and equivalent to cholesterol raft formation within the lipid membrane.

References:


Structure and dynamics of whey proteins covalently modified with cabbage compound allyl isothiocyanate (AITC) at interfaces in disperse systems.

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Keywords: beta-Lactoglobulin, interactions, interfacial tension and dynamics, SAXS

Whey protein isolate (WPI) rich in β-Lactoglobulin (BLG) is frequently used in foods as natural emulsifying agent due to its ability to stabilize oil/water interfaces. However, the interfacial properties are strongly dependent on the pH-value – as at an acidic pH-value the emulsification capacity of WPI is reduced, whereas the foaming capacity is reduced at pH 7.

We found that the covalent attachment of the natural small hydrophobic molecule allylisothiocyanate (AITC) from cabbage to amine- and thiol-groups of BLG in WPI influences its conformation and can increase its hydrophobicity. We hypothesize that this is a promising approach to change the physicochemical properties of WPI in favor of a higher stabilizing effect at interfaces. The overall aim is to increase the emulsifying and foaming stability of WPI over the wide pH-range present in foods.

Different concentrations of the cabbage compound AITC were covalently bound to WPI (rich in BLG) and analyzed by LC-MS. The changed physicochemical properties (zeta-potential, surface hydrophobicity, elasticity and secondary structure) were monitored over a wide pH range (pH 2 to 7) using dynamic light scattering, SAXS, ATR-FTIR and IRRAS to get further insights into the shape, folding and aggregation status of the native and modified proteins at different pH-values. The surface activity of native and modified WPI were evaluated by drop tensiometry and a faster adsorption behavior, as well as lower equilibrium interfacial tension of the modified protein, especially at acidic pH values, were detected.

Rapeseed oil-in-water emulsions were prepared with a high pressure homogenizer at 400 bar using 1 % either modified or unmodified WPI. The emulsifying properties (oil droplet size, creaming stability and information on the interfacial barrier by spin probe analysis) were monitored over a wide pH range (pH 2 to 7) using static light scattering, analytical centrifugation and EPR.

The WPI-AITC conjugates led to emulsions with a significantly smaller droplet size than native WPI at pH 2, a higher creaming stability at pH 2, 4, 6 and 7, and the proteins at the interface were more hydrophobic. Following this, the AITC modification of BLG could be a simple strategy to increase the emulsifying capacity of WPI over a wide pH-range.

References:


Change-over from Linear to Branched Growth During the Nucleation Phase of Lysozyme Crystallization

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Keywords: neutron protein crystallography, crystallization, light scattering, in-situ techniques

Neutron protein crystallography is a powerful tool to investigate enzyme mechanisms in depth. It has the advantage of providing also the hydrogen atom positions which are practically invisible in x-ray crystallography. Furthermore, it does not lead to a reduction of the active center due to radiation damage often encountered using x-rays. Its main disadvantage is the need for rather large crystals (> 0.5 mm\(^3\)). This proves to be the biggest bottle-neck when considering a neutron protein crystallography experiment.

To address this we concentrated on the early stages of the crystallization process where the directions are set whether many small crystals grow or few large ones. We used lysozyme as a model system since it has been studied well in the past and the phase diagram of its crystal growth is known. We used a combination of three scattering techniques since the involved size ranges require a large q-range. Small angle neutron scattering was used in combination with static light scattering on the same sample in order to obtain structural information on the growing crystal seeds. In situ dynamic light scattering at the neutron scattering sample cell was used to obtain an overview of all sizes present in the crystallization process by measuring their hydrodynamic radii. The small angle neutron scattering technique requires the crystallization in heavy water instead of normal water. We found that the crystallization conditions did not differ too much from the ones mentioned in the literature for light water when using a corrected pH value of pD=pH+0.4. The crystallization is initiated by mixing a 60 mg/ml Lysozyme solution with a 6 wt\% NaCl acetate buffer solution (both at pD=4.75 and at 298 K) in a 1:1 ratio. Immediately after mixing, dimers of lysozyme molecules are formed and the structure factor seen in the lysozyme stock solution disappears. Under the chosen conditions we could observe a fractal growth of the crystal seeds with a change of the fractal dimension from 1.0 to 1.7 in the first 90 min. This can be interpreted as a crystal seed being formed first which grows more in a linear manner with little branching. Later, the space in between the branched arms is filled to cross over to a more densely packed fractal. With these results theoretical models of crystal growth can be improved. Furthermore, the early detection of crystal seeds can be used to rapidly change the crystallization conditions (e. g. temperature) in order to avoid the production of more crystal seeds.
Quality and Reproducibility of Tethered Model-Membranes As Seen By Neutron Reflectometry and Complementary Techniques

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Keywords: neutron reflectometry, lipids, membrane, tether, reproducibility

Transport processes through membranes play an important role in many aspects of life: membrane proteins, the interaction of peptides or enzymes with membranes or the transport of drugs into cells are some examples among many others. However, it is still a challenge to investigate these processes. Solid supported bilayers are a well-known system to study the interaction of molecules with model membranes using high-resolution techniques as neutron- and X-ray reflectometry (NR, XRR). A limitation of the use of simple solid supported bilayers is that the membrane proximity to the solid substrate can hinder the penetration of molecules through the membrane. Therefore, a highly hydrated spacer or tether is needed between the solid support and the membrane. The requirements for tethered model-membranes to be used with reflectometry techniques are challenging: (i) low roughness/waviness over a large area, (ii) homogenous over a large area, (iii) stable, (iv) easy to prepare, (v) penetrable for molecules, and (vi) reproducible. In the last ten years a lot of work was devoted to the development of different tethers, like polymer brushes, protein layer or floating bilayer, as well as of different preparation techniques as Langmuir-Blodgett/Langmuir-Schaefer transfer, solvent exchange, vesicles fusion or spin-coating. Here, I will present our recent results using a combination of short and long polymer brushes (polythelyenglycol, PEG) as tether seen with NR, XRR, atomic-force-microscopy (AFM) and fluorescence-microscopy. I will focus on different preparation techniques and discuss the results in aspects of quality and reproducibility.

Figure: Neutron reflectometry and corresponding scattering length density profile in three different contrasts, four times the same experiment: mixture of short and long polymer brushes (PEG) as tether on a silicon bloc, solvent exchange as preparation method for the POPC-lipid bilayer as model-membrane. Contrasts are shifted in respect to each other for clarity. Included sketch (left) was taken from Hertrich et al. Sketch of POPC (right) taken from Avanti Polar Lipids.

References:
Investigation of Molecular Dynamics: Exceed the Gaussian Approximation

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Keywords: elastic neutron scattering, dynamics of biological systems, MD simulations, theoretical models

In 2013, following the ideas which led to the creation of the very successful Protein Data Bank (pdb) for structures, G. Zaccai and coworkers proposed to build up a neutron Dynamics Data Bank (nDDB) which would be populated with already existing and new dynamical data of biological macromolecules measured with different techniques, mainly neutron scattering, NMR and molecular dynamics (MD) simulations. On one hand, this should result in a global picture of the dynamics of biomolecules measured using different instruments (i.e. different dynamical ranges and resolutions) and on the other hand help to combine experimental data and simulations into a common picture. This data base would then be freely accessible by the scientific community. A first step in this direction consisted in a literature survey covering more than 300 papers, which showed that a large fraction of the published results consisted in values for the mean square displacement as a function of temperature, $\langle u^2(T) \rangle$ . Unfortunately, the $\langle u^2(T) \rangle$ values extracted from experiment may depend strongly on the instrument resolution, Q-range and analysis method employed, making difficult to make quantitative comparisons between proteins, but also between experimental data and MD simulations. As it is important to understand these differences, we aim to test current models to analyse in a more consistent way elastic, quasi-elastic and inelastic neutron scattering data and to investigate which is the more appropriate way to compare them to MD simulations’ results. This step is crucial to allow neutron scattering data to be widely used to validate and refine the force fields usually employed in biomolecular simulations. Our goal is to find out how quantitatively MD simulations can reproduce the trends of experimental data over a range of different systems and instruments and under many different conditions like temperature, pressure, hydration, pH/pD or crowding. In addition, it is important to figure out to what extent such trends may depend on sample preparation, data reduction and data analysis.

Generally elastic data is analyzed based on the Gaussian approximation with Q values normally not exceeding 2Å$^{-1}$. Here we present data on a larger momentum transfer range - up to Q=5Å$^{-1}$ as measured on the IN13 spectrometer at the ILL - and investigate the accuracy of different models which have been proposed lately, see e.g. ref. [3-5]. This could point to the extraction of more information from elastic data sets. The aim is to investigate the significance of this additional information with experiments and MD simulations.

References:

Time-of-flight neutron diffraction textures and modeled elastic anisotropies of high pressure rocks form an exhumed subduction channel

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The exhumation of high pressure (HP) metamorphic rocks at converging plate margins can be explained by the existence of a so-called subduction channel, a shear zone of a few kilometer thickness above the downgoing tectonic plate. In this channel material is transported deep into the earths’ mantle and exhumed back to crustal depth. Despite elaborate investigations of paleo-subduction channels, the tectonic processes within these shear zones, particularly the exhumation of HP rocks from depths of up to 80 km, are not completely understood. High resolution seismic imaging of their interior has not been possible so far, which is in part due to a lack of crystallographic preferred orientation (CPO) data of subduction channel rocks and hence, a lack of related elastic anisotropy (EA) data. CPOs of these polyphase crustal rocks are difficult to acquire and data is not widely available.

In this study, CPO analyses of a set of subduction channel rocks were performed at the neutron time-of-flight texture diffractometer SKAT at the Frank Laboratory of Neutron Physics at JINR, Dubna, Russia [1], [2]. Due to the low absorption of neutrons in matter large sample volumes can be measured, which better resemble the bulk texture. This is particularly important for calculation of global physical rock properties. Furthermore, the application of RIETVELD structural refinement allows the determination of crystal structure and texture of polyphase samples [3]. Rock EA was calculated using the Christoffel equation, which combines seismic velocities, single crystal stiffness coefficients and density. The weighted average of the EA of the constituent mineral aggregates is obtained using the Orientation Distribution Function and the corresponding single crystal elastic constants.

Samples were collected in the Eclogite Zone of the Tauern Window, in Austria. This unit comprises HP sedimentary rocks, as well as lenses of eclogites, which represent HP basalts, originating from former oceanic crust. Peak pressure-temperature (PT) conditions of 20-25 kbar and 600 +/- 30°C were reached at 32.8 ± 0.5 Ma (Lu-Hf dating of garnet: [4]) and subsequently exhumed in only a few Ma.

In eclogites, which still comprise the HP mineral assemblage, EA is only up to 1.5%. In samples, which were overprinted by lower PT conditions, a higher EA of about 3% is reached. In the HP sedimentary rocks a pronounced CPO leads to EAs between 5.2 and 7.4%. These results allow conclusions on seismic properties of rocks in subduction zones, as well as deeper structural levels of the Alps. Furthermore, the data obtained in this study allow for numerical simulations of seismic wave propagation, which can be used to seismologically explore the exhumation of rocks within subduction channels.

Residual and Applied Strain Investigation on Sandstone Samples Using Neutron Time-of-Flight Diffraction at the Strain Diffractometer EPSILON

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Keywords: geological material, applied strain, residual strain, neutron-TOF-diffraction

The knowledge of the stress and strain state in rock samples is of importance in the understanding of the rock properties for the interpretation of geodynamic processes and for geotechnical applications such as mining and tunneling. Recently, the topic of induced seismicity has become a major issue in geoenergy production (gas production, geothermal). Thus, the role of tectonic stress for the initiation of rock failure is of high scientific but also economic interest. The investigation of deformation with conventional lab scale experiments allows the determination of macro-strain in the cm- to m-range. On the nano- to micro-scale, the application of diffraction methods offers the investigation of the strain which is localized in the crystal lattice, the so-called intracystalline strain. This localization of strain in a sample with dimensions in cm-range is achieved by strain scanning. Because of the low absorption properties of neutrons in matter, like metals and minerals, neutron diffraction is an excellent method for the investigation of strain in bulk samples, especially in multiphase samples, like rocks.

The neutron time-of-flight strain diffractometer EPSILON operated at the pulsed neutron source IBR-2M at the JINR Dubna is designed for the investigation of residual and applied intra-crystalline strain of bulk samples. Because of the large wavelength-range ($\lambda = 7.8$ Å) diffraction pattern with $d$-spacings up to $d = 5.6$ Å can be investigated. Using the long flight path of about 107 m a very good spectral resolution is achieved. That allows the investigation of multiphase rocks containing minerals with lower crystal symmetry. The diffractometer is equipped with a uniaxial pressure device, allowing load states up to 100 kN, e.g. 150 MPa with sample diameters of 30 mm and 60 mm length<sup>1</sup>.

To investigate the spatio-temporal evolution of failure for cyclic axial loading and unloading of sandstone rock samples, residual and applied strain investigations were carried out in samples of porous Lahr sandstone (Southern Germany). The investigations concentrated on 3 locations within the sample: in the center, at the rim and at a location in-between (half-radius). To avoid immediate rock failure a pre-experiment to determine the failure stress has been performed.

![Figure: Geometry and gauge volume for detection residual strain by neutron diffraction in a sandstone sample.](image)
An Approach to Forward-Model Time-of-Flight Neutron Diffraction Data for Uniaxial Load Conditions Using the Example of Sandstone

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Keywords: Neutron Diffraction, Modeling, Time-of-Flight, Young’s Modulus, Voigt Reuss.

Mechanical properties of (sedimentary) rocks such as stress-strain relations are essential to understand processes within the Earth’s crust, e.g. the dynamic processes in oil and gas reservoirs, underground gas storages or in geothermal energy exploration. The measurement of in situ lattice strains in bulk samples is here realized by high-resolution neutron diffraction. The advantage of neutron diffraction is, among others, the high penetration depth which allows to gather a statistically relevant number of grains of natural samples. Hence, a three-dimensional stress-strain behavior can be determined.

This study deals with load-dependent deformation data of a cylindrical homogeneous sandstone specimen from Southern Germany (Kuhbach/Lahr), examined by time-of-flight neutron diffraction method at the pulsed neutron source IBR-2M of the Joint Institute for Nuclear Research (JINR), Dubna (Russia). In contrast to commonly performed Rietveld refinement, this approach focusses on a prediction of measured diffraction data at elevated uniaxial loads based on the load-free measurement and the known elastic properties of single crystal α-quartz. A spherical elastic-mechanical model, representing quartz grains and describing the stress-strain behavior of the probed quartz sandstone, is presented. It incorporates changes in pore space due to compaction of the sediment by modifying the quartz grains’ shape. This model’s behavior is taken to predict the shift of the diffraction pattern lattice peaks based on the models proposed by Voigt and Reuss and combinations of these. Modeling results for distinct applied uniaxial stresses are compared to measured data.

It is shown that it is possible to forward-model (i.e. predict) diffraction data at elevated uniaxial load-stages based on the load-free measurement by using an appropriate elastic-mechanical model describing the probed specimen. In case of the exemplarily treated sandstone, measured diffraction patterns of three uniaxial load-stages (27.78 MPa, 37.51 MPa, 47.02 MPa) are in good agreement with model predictions using the Voigt model and setting a flattening of the “grains” (spheres) of 6.7 %. The model predicts both well, the variation in full width at half maximum (FWHM) and the shift of the diffraction lines with increasing load. The diffraction pattern forward-model quality is in the range of $R_{wp} \sim 1-2 \%$ (weighted profile $R$-factor).

References
Moving the Frontier in Geoscience by Neutron Experiments

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Keywords: geosciences, neutron, instrumentation, structure, geodynamic

Within the last decades, a lot of information was gained about our outer space by satellite missions and innovative observatories. On the other hand, much less is known about the processes and mechanisms within the interior of our dynamic planet. Even though, this processes and mechanisms shape our planet and are a necessity for life on Earth. Natural events – such as volcanism or earthquakes – have the potential to harm huge amounts of population, especially if we ignore the possible hazards and if we do not look ahead. However, first we need a better understanding of the underlying processes.

Recent Georesearch focuses on processes of natural and induced seismicity and safe use of the underground for energy and water supply, the storage of fuels and as disposal site for waste. Furthermore, the subsurface can be used to reduce humankind’s impact to global warming by storing huge amounts of CO₂ within the underground.

Here we will focus on rheological properties of geomaterials crucial for a fundamental understanding of processes and to better face the challenges mentioned above. There is a variety of experimental setups to investigate the reply of geomaterials to changing geomechanic boundary conditions, such as the creation of new inner surfaces and openings in tunnelling and drilling, reducing or rising pore pressures in porous layers or changing the underground heat exchange volumes in geothermal reservoirs.

For rocks consisting of various, mainly low symmetric, often coarse-grained minerals with a wide variety of compositions neutron experiments open a unique window into the subsurface, as it allows high resolution examinations of natural material where rocks can be examined as a whole. Due to new instrumentations and modelling tools, we can study right now and even more in the near future relevant properties to reduce the risk associated with seismicity.

We will present new insights from neutron experiments and future challenges for studies of
- Induced seismicity, caused while using the underground for hydrocarbon exploitation, geothermal energy, and storage operations
- The possibilities of forward models to better test hypotheses
- High pressure studies to better understand the processes within the deep planetary interior.

References:
Neutron Textures for the Analysis of the Deformation History of Rocks

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Keywords: neutron texture analysis, naturally deformed rocks

The understanding of the kinematics and the usually complex deformation history of rocks from the continental crust is crucial for the understanding and modelling of geodynamic processes. Rock textures are one of the important rock fabric parameters to reveal deformation mechanisms/rheology and deformation histories. For the texture analysis of deformed coarse grained rocks, neutron diffraction is the most suitable method, as only this method allows to measure large sample sizes which yields appropriate grain statistics for grain sizes up to mm-scale. The specifications of the SKAT texture diffractometer at the reactor IBR-2 in Dubna, Russia¹ especially allow to measure large sample volumes up to 50 mm in diameter due to the large beam size and also to measure polyphase samples due to the relative high 2Θ-resolution resulting from the time-of-flight method. Applying the Rietveld-based program MAUD² on the measured texture data, allows quantitative texture analyses of single mineral phase in polyphase composed samples.

Recent analyses of deformed rocks regarding their textural and microstructural development focus on marbles as well as rock salt and metasedimentary rocks.

Some of the marble samples originate from the eastern part of the axial zone of the Pyrenees, France, another set of samples derives from the Erzgebirge, Germany. Both of the marble sample sets are especially interesting in view of the texture analysis as they show large grain sizes and non-deformational microstructures supposed to be induced by post-deformational annealing processes. In these cases, only the crystallographic preferred orientations (textures) are indicating earlier deformation processes controlled by dislocation creep. These results help to understand the regional tectonic development and the role of the marbles within orogenic processes, as carbonate rocks often play a key role as lubricant of shear zones during orogenic processes, due to their high ductility at relative low temperatures.

The rock salt samples originate from salt deposits in northern Germany. Also rock salt fabrics show relative large grain sizes up to mm-scale. Therefore texture measurements are challenging but at the SKAT-goniometer in combination with a special measurement strategy possible. By measuring the sample at different tilt positions, the grid density of measured data points can be increased.

Another case study will present the texture analysis of polyphase rocks for which diffraction peaks are overlapping and therefore increases the difficulties of quantitative texture analysis. Texture analyses of metasedimentary rocks consisting of quartz, feldspar and mica from the Chalkidiki peninsula in northern Greece will be presented.


The Method of Soil Moisture Sensing by Cosmic Ray Neutrons

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Keywords: fast neutron, environment, soil moisture, cosmic-ray, instrumentation

Neutron Scattering instrumentation outside of the laboratory: Large scale quantification of environmental water reservoirs has turned out to be feasible by detecting cosmic albedo neutrons. Such are generated in particle air showers in the upper atmosphere and reflected from the ground in dry conditions or efficiently slowed down in the presence of soil water. As the mean free path of high energetic and fast neutrons can be in the order of 100 meters, the environmental neutron density therefore strongly depends on the water content present around a sensor with a large radius of influence. Here we given an introduction to the method and present Monte Carlo based simulations and measurements to study the footprint for various conditions [1]. We furthermore investigate different contributions to the neutron signal and the influence of spatial structures and environmental conditions [2]. Stationary detectors as well as mobile surveys with the Cosmic-Ray Rover reveal pros and cons of this state-of-the-art technology.

References:
Correlating the Ripening Behaviour of Gold Nanoparticles with the Micelle Structure of the Stabilizer CTAB

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Keywords: gold nanoparticles, CTAB, micelles, SAXS, SANS, Ostwald ripening

Gold nanoparticles (AuNPs) exhibit outstanding optical properties. The plasmonic behaviour of such particles is strongly dependent on their size and shape. The synthesis includes high amounts of the structure directing agent and micelle forming surfactant hexadecyl-trimethyl-ammonium bromide (CTAB).[1] By using a strong reducing agent like NaBH4 very small diameters in the range of a few nanometers can be achieved.[2] These particles can be used to synthesize gold nanorods.[1] But as these small AuNPs tend to age within a few hours to days, they must be prepared freshly before each gold nanorod synthesis. We found that the time stability of these nanoparticles can be extended tremendously by the addition of hexanol. As this alcohol also affects the structure of the CTAB micelles, we were able to correlate the AuNP stability with the micelle morphology. A deep knowledge about the micelle structure is therefore needed because the interfering scattering of AuNPs and micelles thwarts simple solvent scattering subtraction. To clarify the influence of hexanol on the CTAB structure in aqueous solutions we did small angle neutron (SANS) and X-ray (SAXS) scattering experiments on CTAB and CTAB/hexanol solutions, respectively. The simultaneous fitting resulted in ellipsoidal micelles that are much more elongated in the latter case. We noticed that this leads to the formation of slightly larger AuNPs that are more stable against ageing. The SAXS fitting revealed a bimodal particle distribution where the larger fraction grows and the smaller one shrinks with increasing storing time. This behaviour is typical for Ostwald ripening. The amount of larger particles is smaller in the case of the hexanol modified CTAB solutions which indicates a much better stabilizing effect against ripening compared to the unmodified CTAB solutions. The poster will show a systematic SAS study dealing with the influence of different alcohols on the ripening behaviour of AuNPs in aqueous CTAB solutions and will help to explain the stabilizing effect of CTAB micelles on AuNPs.

Fig.1: Simultaneous SANS (recorded at KWS-2, MLZ, Garching, Germany) and SAXS (Beamline I911-4, MAX IV laboratory, Lund, Sweden) fitting of a 0.1 mol/L CTAB (upper left) and 0.1 mol/L CTAB/hexanol (upper right) solution in D2O. The fitting resulted in ellipsoidal CTAB micelles with a few nanometers in size which are more elongated in the presence of hexanol. This leads to the formation of more stable AuNPs against Ostwald ripening which can be extracted from SAXS data measured after 2 (black circles, solid red: fit) and 36 hours (blue circles, solid green: fit) of storing (CTAB: left, below CTAB/hexanol: right, below).

References:
Stabilization of ZnO Quantum Dots Characterized by In Situ Small Angle Scattering

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Keywords: neutrons, X-rays, small angle scattering, nanomaterials characterization

Semiconductor nanoparticles are of special interest in research as their properties can be adjusted due to the quantum size effect which allows many applications in different electronic devices and thin film solar cells.\(^1\) To design stable and well-defined nanoparticles knowledge about the growth processes and the influence of stabilizers is crucial. Standard characterization techniques can determine the size of the nanoparticle core (SAXS, UV/Vis, TEM) but lack in characterizing the stabilization shell. Using SANS information regarding the organic stabilizer can be achieved, but it is the combination of in situ SANS and SAXS, which allows to obtain a deeper understanding of the stabilization due to the different contrasts of the two in situ methods toward quantum dot core and organic shell.

By the simultaneous analysis of SANS and SAXS scattering patterns of the same sample on absolute scale we were able to observe the stabilizing shell of ZnO nanoparticles in their native solution for the first time (Figure 1A+C).\(^2\) Furthermore, we studied the effects of purification of the ZnO nanoparticles and its influence on the shell using this method. In addition the results from ligand exchange reactions with a catecholic stabilizer will be presented. (cf. Figure 1B+D).\(^3\)

Figure 1: SAXS (green open squares) and SANS (blue open circles) data and fit curves of the simultaneous fit for ZnO nanoparticles A) in the native dispersion and B) after ligand exchange with a catecholic stabilizer. The dotted lines in A) show the influence on the fit curve, if the shell is neglected. C) and D) show the stabilization of the ZnO nanoparticles after synthesis and after ligand exchange, respectively.


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**Keywords:** neutrons, X-rays, small angle scattering, nanomaterials characterization
Impact of Moisture on the Morphology of Hybrid Organo-Metal Halide Perovskite Thin Films for Photovoltaic Application

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Keywords: perovskite solar cells, moisture, in-situ GISANS, structure, dynamics

Organo-metal halide perovskites mark a paradigm shift in photovoltaic research, as they combine high power conversion efficiencies with easy processing and cheap, abundant precursor materials making them a promising contender for other photovoltaic technologies. The commercial application of perovskite solar cells, however, is so far impeded by fundamental understanding lacking behind this fast progress in device efficiency. Though progress in device efficiencies has been remarkable, especially the sensitivity of the perovskite towards ambient moisture is a big issue which basically renders them unsuitable for widespread use as toxic lead – contained e.g. in CH₃NH₃PbI₃, the most commonly used hybrid perovskite – could leak into the environment. Therefore, a lot of research focusses on improving the moisture stability of the perovskite, however, the process of the water uptake itself was so far not deeply addressed. Due to the high sensitivity of CH₃NH₃PbI₃ to water, it tends to hydrolyze in the presence of moisture, leading to the degradation of the perovskite at a critical humidity of 55 %rh [1].

Using grazing-incidence small angle neutron scattering (GISANS) and introducing D₂O vapor, we probed the kinetics of water uptake and followed the structural evolution of a perovskite film synthesis with a common method [2]. We applied several humidities including humidities below and above the critical 55 %rh. In the vertical line cuts of the 2D GISANS data the water uptake is apparent from a shift in the Yoneda peak, whereas the horizontal line cuts show clear changes in the low q-region (complete degradation) and slight changes are visible at higher q indicating a shrinking of crystal domains at moderate humidities.

Based on this knowledge routes to stabilize perovskite films against moisture can be developed leading to higher long-term stability and less environmental hazards thus promoting fast market introduction of perovskite solar cells.

References:
Encoding the Shape of Nano-Assemblies: A SANS-Thermodynamics Study as Key to a Fundamental Understanding of Structure Directing Effects

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Keywords: SANS, nanostructure, self-assembly, scattering, polyelectrolyte

A variety of structures and functions in natural systems is realized by supramolecular assemblies, ranging from cell membranes to protein complexes for photosynthesis. Inspired by natural systems, self-assembly is an important route to organizing soft matter. For example, with regard to the world's decreasing energy resources, developing strategies to exploit solar energy become more and more important: one approach is to self-assemble synthetic functional species to form more effective or tailored supramolecular units. Key to establishing such concepts as a route to applications is to fundamentally understand structure directing effects, which can only be tackled by a detailed structural analysis of the novel nano-objects directly in solution: Core here is the structural analysis by small-angle-neutron scattering (SANS).

In this contribution, we show how well-defined nano-objects can be formed through electrostatic self-assembly of macroions and multivalent aromatic counterions: assembly shapes range from spheres and cylinders over vesicles to networks. The resulting supramolecular structures characterized by SANS, via pair distance distribution function (P(r)) analysis and structural model fitting, partly after combining SANS and static light scattering data. Dynamic light scattering and AFM yield complementary information.

The nanostructure analysis is then related to a thermodynamics study of the assembly process. In this study we reveal how the molecular building block structure directs the supramolecular nanoscale structure, in particular the particle size and shape, on a 10-100 nm level. Fundamentally, thermodynamics including a delicate interplay of interaction forces encodes the shape and size of the nano-assemblies. It turns out that the central point is the enthalpy/entropy balance. Thermodynamics, interplay of interaction forces and dye-dye stack formation will be discussed in for the supramolecular structures characterized by SANS. A general relationship of molecular structure, enthalpy/entropy balance and assembly shape will be presented.

Furthermore, size and shape can respond to pH and light, and photocatalytically active structures for solar energy conversion can be built, where again a careful SANS structure analysis is the key to establishing these concepts.

References:
Self assembly and structural properties of magnetic nanoparticles on silicon surfaces

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Neutron reflectometry and small angle neutron scattering were used to study the assembly of magnetite nanoparticles in a water-based ferrofluid. The particles have an average diameter of 25 nm and are solute in a D₂O–H₂O mixture of 85:15. The particles are designed for biomedical applications. They are surrounded by biocompatible shells consisting of polymers for steric repulsion, like e.g. carboxylic acids. The silicon surfaces were treated differently to provide varying wetting conditions for the particles. Information from neutron reflectometry under different magnetic field conditions as well as small angle neutron scattering measurements were combined to extract information about wetting, layering and structural properties of the nanoparticles in the water mixture. The reflectivity data were fitted to a model using a least square routine based on the Parratt formalism. A densely packed wetting layer and a layered structure of the nanoparticles in the vicinity of this wetting layer close to the silicon surface are found. Different behavior was observed in the non-filed case and under a magnetic field of 100 mT applied parallel to the silicon surface. From the fits of the small angle neutron scattering data information about size of cores and shells and polydispersity of the particles in H₂O and D₂O can be extracted. Combined fits also provide information about a dimerization in the ferrofluid. Combining this information with the neutron reflectivity results also helps to better understand the layering at the silicon surface and the differences observed with and without an applied magnetic field.
The Structure of MAPbI₃: a Neutron and Synchrotron X-ray Diffraction Study

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Keywords: neutron, synchrotron, structure, perovskites, MAPbI₃

In recent years hybrid perovskites became of high interest due to their huge variety of element substitutions possibilities on both cation and anion sites and thus the tailoring of new materials for solar energy conversion. Our field of interest lays in methylammonium lead triiodide (MAPbI₃). In this ABX₃ structure type A is the organic unit [CH₃NH₃]⁺, B=Pb²⁺ and X=I⁻. Recent studies revealed that especially the organic molecule influences the structural properties to a critical point but the complexity of structure due to disordering in both, inorganic and organic part make structural determinations challenging¹.

Up to now there are three temperature dependent structural modifications known: the high temperature phase with space group Pm-3m, which undergoes at 327.4 K a structural phase transition to I4/mcm. In this room temperature modification the number of disordered states of the methyl ammonium molecule is lowered from 8 to 4 possible positions². In the orthorhombic (Pmc2₁) low temperature modification (Tc= 162.2K) the orientation of the organic cation is fixed. Baikie et al.¹ stated a temperature dependent hysteresis in the cubic-tetragonal transition.

This study presents an in depth investigation of the crystal structure of MAPbI₃ with a special focus on the position and orientation of the MA⁺ molecule. A polycrystalline perovskite sample was used, which was synthesized from an equimolar mixture of methylammonium iodide (MAI) and PbI₂¹.

For determination of structural details of the orthorhombic, tetragonal and cubic modification of MAPbI₃ neutron and synchrotron experiments were performed at the fine resolution powder diffractometer E9 at the Berlin Research Reactor BER II and at the diffraction end station of the KMC-2 beamline at the synchrotron source BESSY II. With a combined analysis performed by Fullprof Suite and F.O.X. it was possible to finally determine the correct position and anisotropic displacement parameters of the molecule in the crystal structure. The use of rigid body modelling for the methylammonium ion results in a precise description of the hydrogen positions in the structure, which is superior to previously published structure solutions. This allows a meaningful analysis of the pattern of hydrogen bonds and the related distortions of the structure³.

References:
Lattice Thermal Expansion of Bi$_2$Fe$_4$O$_9$, Bi$_2$Fe$_2$Mn$_2$O$_{10}$ and Bi$_2$Mn$_4$O$_{10}$

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Keywords: neutron diffraction, thermal expansion, magneto-volume effect,

Both Bi$_2$Fe$_4$O$_9$ and Bi$_2$Mn$_4$O$_{10}$ were reported as multiferroics although their centrosymmetric crystal structures are not typical for ferroelectric behavior as the dipoles of each constituent polyhedra orient in different directions. We report the temperature-dependent nuclear and magnetic structures of Bi$_2$Fe$_4$O$_9$, Bi$_2$Mn$_4$O$_{10}$ and Bi$_2$Fe$_2$Mn$_2$O$_{10}$, each of them conforms to the mullite-type structures [1]. The structural and vibrational features were studied using time-of-flight neutron powder diffraction between 10 K and 300 K and Raman spectroscopy. The nuclear and the magnetic structures of Bi$_2$Fe$_4$O$_9$ and Bi$_2$Mn$_4$O$_{10}$ agree well with those of early studies. Temperature-dependent structural data provide possible ferroelectric displacements and magnetic features (moment, structure, couplings) as well as their thermodynamic behaviors. Whether the ferroelectric property is related to the stereochemical activity of the 6s$^2$ lone electron pairs of Bi$^{3+}$ the Liebau density vector [2] and the Wang-Liebau eccentricity parameter of the Bi$^{3+}$O$_6$ octahedra were calculated and compared. Beside considerable conflicts on the structure of Bi$_2$Fe$_2$Mn$_2$O$_{10}$ we determined its nuclear and magnetic structures. Neutron probe clearly distinguishes between Fe and Mn in the M$^{3+}$O$_5$ site due to contrasting scattering lengths. Rietveld analyses showed that the cell parameters lie close the respective mean value of the end-members. The EDX results showed a Mn/Fe ratio of 1:1, and both the Raman and the FTIR spectra show complementary compound-bands produced from two end-members. The lattice thermal expansion was simulated using Grüneisen first-order approximation for the zero pressure equation of state, where the internal energy was calculated by Debye-Einstein-Anharmonicity model [3-5]. In this model the Grüneisen parameter accounts for the isothermal anharmonicity while a low perturbed quantum anharmonicity term measures the isochoric anharmonicity. The characteristic frequencies extracted from the lattice thermal expansion agree well with those calculated from the averaged mass-weighted values of the isotropic atomic displacement parameters. Temperature-dependent magnetic moment was scaled to fit the magneto-volume regions of the respective cell volumes.

References:

Overview of Single crystal diffraction studies with HEiDi

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Keywords: neutron, instrumentation, structure, single crystal diffraction

The single crystal diffractometer HEiDi at the research neutron source Heinz Maier-Leibnitz Zentrum (MLZ) is operated by RWTH Aachen and JCNS. It uses hot neutrons for diffraction studies on various hot topics in different scientific areas related to crystallography like physics, chemistry and mineralogy. Especially in the energy sector and information sector measurements at HEiDi provide users with valuable information about ionic conductors, superconductors and a manifold of magnetic systems by offering high flux, high resolution and large q range respectively, low absorption and high sensitivity for light elements.

For instance, various multiferroic compounds from the melilite family like Ba2CoGe2O7 and Ca2CoSi2O7 have been studied in order not only to reveal their orthorhombic antiferromagnetic order structures in detail at low temperature but also the likelihood of misinterpretation of structure data by multiple scattering effects [1, 2].

High temperature studies on Nd2NiO4+δ and Pr2NiO4+δ have been performed as these brownmillerite structures are today considered as the most promising candidates for mixed electron/ion oxygen conductors at moderate temperatures. The investigation of their oxygen diffusion pathways reveals a double well potential pointing towards the interstitial vacancy sites, creating a quasi-continuous shallow energy diffusion pathway between apical and interstitial oxygen sites [3].

Various studies on silicate and phosphate-based gems show a variety of hydrogen bonds with different degrees of disorder, e.g. in brazilianite, hureaulite and axinite [4, 5, 6].

This contribution will give an overview of the instrument and examples of the aforementioned scientific areas.

Neutrons on the Web - the KFN website "sni-portal.de/kfn"

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Keywords: website, PR, KFN, neutron users

The KFN-website "sni-portal.de/kfn" alias “neutronenforschung.de” is part of the “sni-portal” on research with synchrotron radiation, neutrons and ion beams at large facilities.

Neutron users in Germany are the main target audience of this website, since they elect the KFN every three years as their representation of interests. The website is a platform for communication between users and KFN. Users can register for election and/or for the monthly email-newsletter. They find minutes from the KFN-meetings and news connected to research politics, research, events, proposal deadlines, job offers and much more. On the other hand, users are welcome to contribute to the contents of the website, e.g. by presenting their work as a scientific highlight, by sending in job offers or other news. Just drop me a line at kfnadmin@sni-portal.de! This helps keeping the website alive and interesting not only for other neutron users, but also for potential users and the interested public.

In the near future, a major technical relaunch is planned, so feedback on your experience with the website as well as suggestions for new contents and formats are highly welcome.

References: