



**SNI2018:**

# **German Conference for Research with Synchrotron Radiation, Neutrons and Ion Beams at Large Facilities**

Garching, Sept. 17<sup>th</sup>-19<sup>th</sup>, 2018

# MONDAY

# TUESDAY

# WEDNESDAY

***Instructions:***

*Just click on the name of the day you like to see.*

*Tables of the day are linked to the sessions.*

*Tables of sessions are linked to the abstracts.*

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## Monday – September 17<sup>th</sup>, 2018

Each field is linked to the session's start page. Just click on the title!

<b>09:00</b>	Welcome			<b>2001</b>
<b>09:15</b>	<b>Plenary Talk</b> In situ neutron powder diffraction for energy materials <i>H. Kohlmann</i>			<b>2001</b>
<b>10:00</b>	<b>Keynote</b> Operando XAS investigations on multi-metallic nanomaterials for electrocatalytic applications <i>M. Özaslan</i>			<b>2001</b>
<b>10:30</b>	Coffee break			
<b>11:00</b>	<b>PS 1</b> Instrumentation and methods  <div style="text-align: right;"><b>2001</b></div>	<b>PS 6</b> Nanomaterials and nanostructures  <div style="text-align: right;"><b>0001</b></div>	<b>MS 1</b> In-situ and in-operando studies with special focus on energy materials and catalysis  <div style="text-align: right;"><b>1801</b></div>	<b>MS 2</b> Structural biology: Instrumentation and methods, current trends and topics  <div style="text-align: right;"><b>0350</b></div>
<b>12:30</b>	Lunch break			
<b>14:00</b>	<b>Keynote</b> Secondary neutral and ion mass spectrometry with swift heavy ion projectiles <i>A. Wucher</i>			<b>2001</b>
<b>14:30</b>	<b>PS 2</b> Soft Matter  <div style="text-align: right;"><b>2001</b></div>	<b>PS 5</b> Thin films, 2D and surfaces  <div style="text-align: right;"><b>0001</b></div>	<b>MS 1</b> In-situ and in-operando studies with special focus on energy materials and catalysis  <div style="text-align: right;"><b>1801</b></div>	<b>MS 2</b> Structural biology: Instrumentation and methods, current trends and topics  <div style="text-align: right;"><b>0350</b></div>
<b>16:00</b>	Coffee break			
<b>16:30</b> - <b>18:00</b>	<b>Poster session 1</b>			

<b>19:00</b>	<b>Public lecture</b> Materie durchblickt – durch Synchrotronstrahlung, Röntgenlaserblitze und Neutronenwellen <i>T. Salditt</i>	<b>2001</b>
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## In situ neutron powder diffraction for energy materials

Monday, 09:15  
MW2001

ID 430

**KOHLMANN, Holger (Universität Leipzig)**

Solid materials play a crucial role for many energy-related applications. In situ diffraction is an important method for investigating their synthesis as well as their function and neutrons are an ideal probe. Many energy-related materials contain light elements such as H, Li, N, O, which can often be better located in crystal structures by neutrons as compared to X-rays. Control over external parameters such as pressure and temperature often requires bulky sample environment, which is easier to penetrate by neutrons than by X-rays in general. Further, neutrons can probe many properties of materials, e.g. crystal structure, diffusion, magnetism, vibrations, and thus yield extensive information. In this contribution, we will show that unraveling basic steps of reactions in functional materials under close to operating conditions by time-resolved in situ investigations is the key to rationalize and gain better control over synthesis procedures and over function. Examples include hydrides, oxides and intermetallics as functional materials for rechargeable batteries, fuel cells, hydrogen storage, permanent magnets, and photocatalysis. As the potential of in situ neutron diffraction is intimately connected to instrumental development, recent advances in sample environment will be reviewed.

[1] A. Götze, H. Auer, R. Finger, T. C. Hansen, H. Kohlmann, Phys. B, doi.org/10.1016/j.physb.2017.11.024

[2] V. K. Peterson, J. E. Auckett, W.-K. Pang, IUCrJ 2017, 4, 540



## Operando XAS investigations on multi-metallic nanomaterials for electrocatalytic applications

Monday, 10:00  
MW2001  
ID 288

*ÖZASLAN, Mehtap (Carl von Ossietzky University, Oldenburg)*

Improvements in activity, selectivity and durability of catalytically active electrode materials are of large interest to boost the application of renewable and clean energy conversion and storage systems like polymer electrolyte fuel cells and electrolyzers. Generally, electrochemical reactions like oxygen reduction/evolution reaction or electrochemical CO<sub>2</sub> reduction reaction can be seen as complex multiple proton-electron reactions. In most cases, the relationship between structure and activity as well as the electrochemical processes of these electrocatalysts are poorly understood to date. Therefore, operando studies are needed to design efficient and robust electrocatalysts under realistic operating conditions. In this talk, I will give several examples to uncover the electrochemical processes on multi-metallic nanomaterials for the oxygen reduction reaction and CO<sub>2</sub> reduction reaction probed by in-situ and operando XAS technique. In combination of unique and advanced characterization techniques, we will provide fundamental insights into the reaction mechanisms and kinetics of electrochemical processes on multi-metallic nanomaterials. This knowledge helps to improve the design of efficient and robust electrocatalyst materials for the applications of fuel cells and electrolysis.



## Secondary Neutral and Ion Mass Spectrometry with Swift Heavy Ion Projectiles

Monday 14:00  
MW2001  
ID 415

*WUCHER, Andreas (Universität Duisburg-Essen)*

Mass spectrometry of atoms and molecules desorbed from a solid surface under bombardment with energetic particles provides a versatile method for chemical surface and in-depth analysis. Typical commercial applications involve the use of keV projectile ions and analyze the flux of electrically charged secondary particles (Secondary Ion Mass Spectrometry SIMS). The collisional sputtering process initiated by such projectiles, however, often generates a significant amount of chemical damage both at the surface and in the sputtered material, so that it has been suggested to utilize the electronic sputtering process initiated by a swift heavy ion (SHI) impact in order to minimize molecular fragmentation ("MeV-SIMS"). Moreover, the vast majority of the sputtered material is generally emitted in a neutral state, thereby asking for a suitable post-ionization method in order to render these particles accessible to mass spectrometric analysis (Sputtered Neutral Mass Spectrometry SNMS). We have recently implemented a new time-of-flight mass spectrometer at the GSI UNILAC beam line which enables a combined SIMS/SNMS analysis of material sputtered under SHI impact. First applications of this system have delivered a wealth of data regarding the composition and charge state of the sputtered material, thereby providing valuable insight into the SHI-induced sputtering process. The data also indicate a nearly fragment-free desorption of intact neutral parent molecules from selected SHI-irradiated organic films.



## Materie durchblickt - durch Synchrotronstrahlung, Röntgenlaserblitze und Neutronenwellen

Monday, 19:00  
MW2001  
ID 450

*SALDITT, Tim (Institute for X-Ray Physics, University of Göttingen)*

Röntgenstrahlen und Neutronen durchdringen Materie und liefern so Einblick in ihren atomaren und molekularen Aufbau. Täglich nutzen wir die Erkenntnisse, die daraus gewonnen werden. Diese Strukturanalyse von Materie mit Röntgenstrahlen und Neutronen beruhen zu einem wesentlichen Teil auf Beugungsmethoden, die allerdings nur indirekt und nur durch Mittelung über viele Atome oder Moleküle Ausschluss über deren Struktur geben kann, und das auch nur, wenn die Anordnung einer gewissen „Regelmäßigkeit“ entspricht. Unser Wissen bleibt also gewissermaßen immer auf eine „übermäßig idealisierende Ordnung“ angewiesen.

Durch hochbrillante Röntgenstrahlung aus Synchrotronquellen und Röntgenlasern ist es nun erstmals möglich, Nanostrukturen direkt abzubilden, also ohne Mittelung und für beliebige Anordnung. Damit lässt sich insbesondere auch biologische Materie besser verstehen, zum Beispiel der biomolekulare Aufbau von Zellen und Geweben. Ein besonderer Vorteil besteht ferner darin, dass man scharfe Bilder in allen drei Dimensionen erhalten kann, also Struktur nicht nur „oberflächlich“ betrachtet. Durch kurze Röntgenblitze kann man manche Nanostrukturen sogar „filmen“. Gleichzeitig lässt sich die Bewegung von Molekülen durch Fortschritte der Neutronenforschung immer besser und schärfer beobachten. In diesem Vortrag wird verständlich gemacht, wie solche Experimente im Prinzip funktionieren und neue Erkenntnisse liefern können.



**Monday – September 17<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Parallel session 1***

### ***Instruments and methods***

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>11:00-11:15</b> <i>Christian Gorges</i> Ultracold neutron sources and their applications at the research reactor TRIGA Mainz
<b>11:15-11:30</b> <i>Tobias Neuwirth</i> The upgraded neutron grating interferometer at ANTARES – Design, Performance and Applications
<b>11:30-11:45</b> <i>Michael Lerche</i> The ODIN Project at the European Spallation Source
<b>11:45-12:00</b> <i>Elias Hamann</i> Dose efficient, high resolution X-ray imaging applied to biological model organisms
<b>12:00-12:15</b> <i>Andreas Schropp</i> High-Resolution Scanning X-Ray Microscopy at PtyNAMI
<b>12:15-12:30</b> <i>Jörg Grenzer</i> Ultra-high-speed X-ray imaging of laser-driven shock into solid materials using synchrotron light



## Ultracold neutron sources and their applications at the research reactor TRIGA Mainz

Monday, 11:00  
MW2001  
ID 401

**GORGES, Christian (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz)**

*FREI, Andreas (Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), Technische Universität München); GEPPERT, Christopher (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz); HEIL, Werner (Institut für Physik, Johannes Gutenberg-Universität Mainz); HOLLERING, Alexander (Physik Department E18, Technische Universität München); KAHLENBERG, Jan (Institut für Physik, Johannes Gutenberg-Universität Mainz); KARCH, Jan (Institut für Physik, Johannes Gutenberg-Universität Mainz); PAUL, Stefan (Physik Department E18, Technische Universität München); REICH, Tobias (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz); RIES, Dieter (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz); ROSS, Kim (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz); TRAUTMANN, Norbert (Institut für Kernchemie, Johannes Gutenberg-Universität Mainz)*

The inherently safe research reactor TRIGA Mainz is able to produce neutron pulses with an energy of 10 MWs for a duration of 30 ms. This makes it a perfect tool for the investigation of the free neutron's lifetime and for fundamental neutron research in general. Within the Cluster of Excellence PRISMA, a superthermal source for ultracold neutrons (UCN) was built and is used in pulsed operation at the TRIGA Mainz. A UCN density of up to 8.5 UCN per cm<sup>3</sup> per neutron pulse in a volume of 32 liters was established [1]. In close collaboration with TU Munich, a second superthermal UCN source is operational and can be used in the continuous operation mode with 100 kW thermal reactor power, e.g., to test and improve experimental components. After a short introduction of the TRIGA Mainz reactor, the talk will concentrate on the production mechanism of UCN, the UCN sources and examples of the research activities with UCN in Mainz.

[1] J. Kahlenberg et al., Eur. Phys. J. A (2017) 53: 226



## The upgraded neutron grating interferometer at ANTARES – Design, Performance and Applications

Monday, 11:15  
MW2001  
ID 297

**NEUWIRTH, Tobias (Heinz Maier-Leibnitz Zentrum, TU München)**

*GUSTSCHIN, Alex (Chair of Biomedical Physics, Department of Physics and Munich School of Bioengineering, TU München); BACKS, Alexander; PFEIFFER, Franz (Chair of Biomedical Physics, Department of Physics and Munich School of Bioengineering, TU München); BÖNI, Peter (Physikdepartment E21, TU München); SCHULZ, Michael (Heinz Maier-Leibnitz Zentrum, TU München)*

Neutron grating interferometry (nGI) is a relatively new neutron imaging technique, which simultaneously delivers information about the transmission (TI), phase shift (DPC) and the scattering (DFI) inside a sample [1]. In particular the DFI has generated high interest, due to its ultra-small-angle neutron scattering (USANS) contrast mechanism, allowing to indirectly resolve structures not directly resolvable by an imaging instrument [2]. Recently there have been strong efforts to use nGI and particularly the DFI as tools for quantitative measurements of structures in materials. A prerequisite for such measurements is a high signal-to-noise-ratio. For the DFI it has been shown that the main reasons for high noise are (i) low DFI signal and (ii) low visibility [3]. Hence a high visibility is needed for quantitative measurements. We will present the upgraded nGI setup at the ANTARES beamline at FRM II, which has been heavily redesigned compared to its precursor [4]. The redesign allowed to optimize the source and analyzer gratings. With these changes we have achieved a visibility of 75% over the whole detector area (76mm x 76mm) at the design wavelength of 4 Å. This visibility is close to the theoretical limit imposed by the spatial coherence generated by the G0 grating.

[1] C. Grünzweig, PhD thesis (2009)

[2] M. Strobl et al., 101, 123902 (2008)

[3] R. Harti et al., Review of Scientific Instruments 88, 103704 (2017)

[4] T. Reimann et al., J. Appl. Cryst. 49, 1488-1500 (2016)



## The ODIN Project at the European Spallation Source

Monday, 11:30  
MW2001  
ID 272

*LERCHE, Michael*

*MORGANO, Manuel (Paul Scherrer Institut); CALZADA, Elbio; STROBL, Markus (Paul Scherrer Institut); SCHULZ, Michael; SCHILLINGER, Burkhard*

ODIN (Optical and Diffraction Imaging with Neutrons) is a beamline project at the European Spallation Source (ESS). It is a collaboration between the ESS, PSI and TUM, with TUM as lead institution. ODIN will provide a multi-purpose imaging capability with spatial resolutions down to the  $\mu\text{m}$  range. The pulsed nature of the ESS source will give access to wavelength-resolved information. Different imaging techniques, from traditional attenuation-based imaging to advanced dark field, polarized neutron or Bragg edge imaging, will be available within the full scope of ODIN with unprecedented efficiency and resolution. A summary of the technical full scope and its science application will be given and the updated conceptual instrument design including its challenges, will be presented.



## Dose efficient, high resolution X-ray imaging applied to biological model organisms

Monday, 11:45  
MW2001  
ID 358

**HAMANN, Elias (Karlsruhe Institute of Technology)**

*Baumbach, Tilo (Institute for Photon Science and Synchrotron Radiation (IPS), Karlsruhe Institute of Technology (KIT)); BELLUCCI, Valerio (Laboratory for Application of Synchrotron Radiation (LAS), Karlsruhe Institute of Technology); HESSDORFER, Holger (Institute for Photon Science and Synchrotron Radiation (IPS), Karlsruhe Institute of Technology (KIT)); ENGELHARDT, Sabine (Centre for Organismal Studies (COS) Heidelberg; Institute for Photon Science and Synchrotron Radiation (IPS), Karlsruhe Institute of Technology (KIT)); ZUBER, Marcus (Laboratory of Applications of Synchrotron Radiation (LAS), Karlsruhe Institute of Technology); HOFMANN, Ralf (Institute for Photon Science and Synchrotron Radiation (IPS), Karlsruhe Institute of Technology (KIT)); VAGOVIC, Patrik (DESY, XFEL); HRIVŇAK, Stanislav (Department of Biophysics, Faculty of Science, P. J. Šafárik University, Košice); THUMBERGER, Thomas (Centre for Organismal Studies (COS) Heidelberg); WITTBRODT, Joachim (Centre for Organismal Studies (COS) Heidelberg)*

The main motivation of our work is to develop high-throughput as well as dose efficient X-ray imaging instrumentation and techniques for small animal imaging of vertebrate model organisms with high resolution and adjustable field of view (FOV) for multi-scale observations of whole organisms, organs, and cellular processes. High-resolution 3D and 4D X-ray imaging of model organisms and their developmental processes provides valuable insights and important information for life sciences without the need for dissecting the specimen, thus also allowing for in vivo studies. However, especially in the case of synchrotron in vivo experiments, the dose impinging on the specimen is crucial and has to be minimized. Methodical routes for dose-efficient in vivo studies enclose in-line phase contrast imaging (PCI) and the use of Bragg Magnifier (BM) optics. By using asymmetric Bragg reflection, we achieve a magnification of up to 200 in 2D. By placing the BM downstream of the sample and combining it with a photon counting pixel detector, we obtain a highly resolving (sub- $\mu\text{m}$ ) and very dose efficient X-ray microscope. By placing the BM upstream of the sample, we can adjust the FOV up to several  $\text{cm}^2$  and preserve or even enhance the coherence properties of synchrotron beamlines. In this contribution we show exemplary results of high-throughput imaging of whole Medaka, in vivo PCI measurements of *Xenopus* embryos, as well as the design and first experimental results of the BM instrumentation.



## High-Resolution Scanning X-Ray Microscopy at PtyNAMI

Monday, 12:00  
MW2001  
ID 346

**SCHROPP, Andreas (Deutsches Elektronen-Synchrotron DESY)**

*BOTTA, Stephan (Deutsches Elektronen-Synchrotron DESY); DÖHRMANN, Ralph (Deutsches Elektronen-Synchrotron DESY); FALKENBERG, Gerald (Deutsches Elektronen-Synchrotron DESY); GARREVOET, Jan (Deutsches Elektronen-Synchrotron DESY); KAHNT, Maik (Deutsches Elektronen-Synchrotron DESY); LINDEMANN, Hendrik (Deutsches Elektronen-Synchrotron DESY); LYUBOMIRSKIY, Mikhail (Deutsches Elektronen-Synchrotron DESY); SCHOLZ, Maria (Deutsches Elektronen-Synchrotron DESY); SEIBOTH, Frank (Deutsches Elektronen-Synchrotron DESY); SEYRICH, Martin (Deutsches Elektronen-Synchrotron DESY); WITTEWER, Felix (Deutsches Elektronen-Synchrotron DESY); SCHROER, Christian (DESY / Uni. Hamburg)*

The group 'X-Ray Nanoscience and X-Ray Optics' at DESY and Universität Hamburg works on the development of refractive X-ray optics with optimum performance as well as scanning X-ray microscopy techniques in the hard X-ray regime. These are powerful scientific tools for structure determination that is applied in various scientific fields, such as biology, chemistry (catalysis), physics, materials science and nanotechnology. The Ptychographic Nano-Analytical Microscope (PtyNAMI) installed in the nanohutch of beamline P06 at PETRA III has been developed during the last two BMBF funding periods in collaboration with the Technische Universität Dresden. It is designed to create focused X-ray beams with sizes of 50 nm (FWHM) and even smaller, which allows one to image objects with high spatial resolution yielding local elemental, chemical and structural information of a specimen. Scanning coherent X-ray microscopy (ptychography) can be combined with tomographic methods potentially yielding structural information in 3D with a spatial resolution of 10 nm and even below. The new microscope is mechanically optimized in view of its stability and residual sample vibrations are controlled using optical interferometers, reducing imaging artifacts related to long-term drifts and higher-frequency vibrations of a sample. In this contribution, we present recent results of high-resolution X-ray imaging experiments obtained with PtyNAMI combining fluorescence and coherent diffraction contrast.



## Ultra-high-speed X-ray imaging of laser-driven shock into solid materials using synchrotron light

Monday, 12:15  
MW2001  
ID 140

**GRENZER, Jörg (Helmholtz-Zentrum Dresden-Rossendorf e. V.)**

*OLBINADO, Margie P. (European Synchrotron Radiation Facility); RACK, Alexander (European Synchrotron Radiation Facility); DE RESSEGUIER, Thibaut (Institut Pprime, CNRS - Université de Poitiers ISAE-ENSMA); DANILEWSKY, Andreas (Kristallographie, Institut für Geo- und Umweltwissenschaften); KRAUS, Dominik (Helmholtz-Zentrum Dresden-Rossendorf e. V.); COWAN, Thomas (Helmholtz-Zentrum Dresden-Rossendorf e. V.)*

A high-power, pulsed laser impacting the surface of a material can generate surface ablation, shock waves and crack propagation; while X-ray imaging can provide a time-resolved probe. Hard X-rays are perfectly suitable for visualizations of transient processes in optically opaque materials even for objects of several mm in size. The MHz pulsed time structure, tunable energy bandwidth, high brilliance, and the high degree of spatial coherence of hard X-rays ( $E > 30\text{keV}$ ) from third generation synchrotron sources allowing transient processes to be tracked directly using ultrahigh-speed image acquisition systems. We report on an in-situ real time investigation of ns single-pulsed laser-driven processes studied by combined diffraction-direct-space-imaging experiments exploiting the single bunch structure at the hard X-ray imaging beam line ID19 of the ESRF investigating the process of laser hole drilling into single crystalline silicon. Whereas macroscopic changes in bulk materials can be quite easily deduced from X-ray phase contrast imaging; information probing changes at the lattice level can be obtained using diffraction imaging. The whole process was followed for 120sec with a maximum frame rate of up to 100kHz. We have developed an experimental method that allows to synchronize on the ns level the single shot laser operation with the high speed camera system matching the 4- and 16-bunch structure of the ESRF.



**Monday – September 17<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Parallel session 6***

# ***Nanomaterials and nanostructures***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:00-11:15**

*Carsten Ronning*

In operando x-ray imaging of nanoscale devices: Composition, valence, and internal electrical fields

**11:15-11:30**

*Sabrina Thomä*

Pushing PDF experiments for weakly scattering samples – tackling solvation shells

**11:30-11:45**

*Werner Egger*

Cellulose Nanofibrils Films: Molecular Diffusion through Elongated Sub-Nano Cavities

**11:45-12:00**

*Maria Eugenia Toimil-Molares*

Nanochannels, nanowires, and nanotubes fabricated by ion-track nanotechnology

**12:00-12:15**

*Steffen Tober*

Diffusion of iron in the near-surface region of magnetite (001)

**12:15-12:30**

*Tilo Baumbach*

In-situ X-ray diffraction during molecular beam epitaxy growth of III-V semiconductor nanowire structures



## In operando x-ray imaging of nanoscale devices: Composition, valence, and internal electrical fields

Monday, 11:00  
MW0001  
ID 97

**RONNING, Carsten (Friedrich-Schiller-Universität Jena)**

*JOHANNES, Andreas (ESRF); SALMON, Damien (ESRF); MARTINEZ-CRIADO, Gema (UA Madrid); GLASER, Markus (TU Wien); LUGSTEIN, Alois (TU Wien); RONNING, Carsten (Friedrich-Schiller-Universität Jena)*

We introduce a method for directly imaging depletion layers in operando with elemental specificity and chemical speciation at sub-100 nm spatial resolution applicable to today's three-dimensional electronic architectures. These typically contain complex, multicomponent designs consisting of epitaxial heterostructures, buried domains, or nanostructures with different shapes and sizes. Although the variety of devices is immense, they commonly rely on carrier separation in a built-in potential induced by composition or strain gradients. To image these, we scanned a focused synchrotron x-ray nanobeam over a single semiconductor nanowire heterostructure and simultaneously measured the current through the device and the emitted characteristic x-rays as a function of the incoming hard x-ray energy. With these results, it is possible to identify the compositional and molecular structure as well as localize the electrical fields present under typical working conditions. This information allows us to draw an energy band diagram consistent with the elemental distribution and a high-resolution chemical speciation map.



## Pushing PDF experiments for weakly scattering samples – tackling solvation shells

Monday, 11:15  
MW0001  
ID 91

**THOMÄ, Sabrina (Universität Bayreuth)**

ZOBEL, Mirijam (University Bayreuth); ECKARDT, Mirco (Universität Bayreuth); THOMÄ, Sabrina (Universität Bayreuth)

The pair distribution function (PDF) technique exploits the total, i.e. the Bragg and diffuse, scattering of crystallographically challenging materials to derive their structure - with nanoparticles (NPs) being the biggest showcase in recent years. [1] The strong increase in flux at high X-ray energies combined with novel detector technologies at synchrotron radiation facilities allowed to push both the time resolution of in-situ studies and the concentrations to new, low borderlines. [2,3] Recently, we proved the existence of nanoscopic solvation shells around colloidal NPs < 10 nm in various solvents (alcohols, hexane). [3] The solvent molecules reorient over 3 to 5 molecular layers away from the NP surface before bulk properties are regained. Such insight requires caution in data treatment as slight flatfield imperfections or insufficient polarization corrections have the same contribution to the overall scattering signal as the restructured solvent. Our approach to analyse double-difference PDFs of restructured solvent layers is now readily applied to colloidal iron oxide NP dispersions in organic solvents, nanodiamonds in water and palladium NPs in tetrahydrofuran. Novel focus is on the interfacial solvent structures in liquid-phase catalysis as well as extending the measurable temperature and NP size range.

[1] S. J. L. Billinge, et al., Chem. Commun. 7 (2004), 749

[2] M. Zobel, et al., CrystEngComm 18 (2016) 2163

[3] M. Zobel, et al., Science 347 (2015), 6219, 292

## Cellulose Nanofibrils Films: Molecular Diffusion through Elongated Sub-Nano Cavities

Monday, 11:30  
MW0001  
ID 395

**EGGER, Werner (Institut für Angewandte Physik und Messtechnik, Universität der Bundeswehr München)**

*ROILO, David (IdEA Laboratory, Department of Physics, University of Trento); MAESTRI, Cecilia (Nanoscience Laboratory, Department of Physics and Centre for Integrative Biology (CIBIO), University of Trento); SCARPA, Marina (Nanoscience Laboratory, Department of Physics, University of Trento); BETTOTTI, Paolo (Nanoscience Laboratory, Department of Physics, University of Trento); KOSCHINE, Toenjes (Institut für Angewandte Physik und Messtechnik, Universität der Bundeswehr München); BRUSA, Roberto (Department of Physics, University of Trento and INFN-TIFPA, University of Trento); RICCARDO, Checchetto (IdEA Laboratory, Department of Physics, University of Trento)*

Packaging technology is currently based on the use of petroleum-derived polymer materials. Environmental concerns, production costs and end-of-life disposal challenges require the introduction of innovative packaging materials produced using green technologies and able to meet requirements such as biodegradability and carbon neutrality. Cellulose nanofibrils (CNF) are highly stable nanostructures obtained from cellulose pulp that show excellent optical and mechanical properties. CNF films and CNF surface coatings attract great technological interest in order to replace the petroleum-based commercial packaging materials. Gas-phase permeation measurements show that few nanometers thick CNF films are impermeable barriers for CO<sub>2</sub>, O<sub>2</sub>, and N<sub>2</sub> but permit the selective transport of 2H<sub>2</sub> and He. Diffusive paths consist of interconnected elongated cavities between tightly packed cellulose nanofibrils. Depth-profiled positron annihilation lifetime spectroscopy measurements in the 1–12 keV positron implantation energy range with the pulsed low energy positron beam (PLEPS) at the MLZ in Garching indicate a cavity size of ~0.31 nm, suggesting that the selective transport of small penetrants is due to sieving effects. Diffusion has configurational character and occurs by thermally activated process with  $39 \pm 1$  and  $33 \pm 2$  kJ mol<sup>-1</sup> activation energy for 2H<sub>2</sub> and He, respectively.

Roilo et al., J. Phys. Chem. C 121 (2017) 15437–15447, DOI:10.1021/acs.jpcc.7b02895



## Nanochannels, nanowires, and nanotubes fabricated by ion-track nanotechnology

Monday, 11:45  
MW0001  
ID 246

**TOIMIL-MOLARES, Maria Eugenia (GSI Helmholtz Center)**

*ULRICH, Nils (GSI Helmholtz Center); YANG, Florent (GSI Helmholtz Center); SCHUBERT, Ina (GSI Helmholtz Center); DISTEFANO, Giuseppa (GSI Helmholtz Center); WAGNER, Michael (GSI Helmholtz Center); TRAUTMANN, Christina (GSI Helmholtz Center)*

This talk will illustrate how swift heavy ion-track nanotechnology provides an excellent platform (i) to fabricate tailored nanochannel sensors, (ii) to fabricate nanowires and investigate their sizedependent properties, and (iii) to develop 3-D and multicomponent nanostructure assemblies. Membranes with parallel nanochannels are fabricated by swift heavy ion irradiation and subsequent chemical etching. In addition, templates with interconnected tilted nanochannels are obtained by applying ion irradiation at several incident angles in consecutive steps. Nanochannel density and orientation, as well as diameter and geometry, are adjusted by the irradiation and etching conditions, respectively. Subsequent electrodeposition in the channels results in nanowire arrays and highly ordered 3-D nanowire ensembles of various materials. Recent developments achieved on the electrodeposition of metal (Au, Cu), semiconductor (ZnO and p-Cu<sub>2</sub>O) and semimetal (Sb) nanowire arrays and nanowire networks will be also presented.



## Diffusion of iron in the near-surface region of magnetite (001)

Monday, 12:00  
MW0001  
ID 285

**TOBER, Steffen (DESY / Universität Hamburg)**

*LOTT, Dieter (Helmholtz-Zentrum Geesthacht); VONK, Vedran (DESY Nanolab); CREUTZBURG, Marcus (DESY / Universität Hamburg); SYED MOHD, Amir (Juelich Centre for Neutron Science JCNS); PÜTTER, Sabine (Jülich Centre for Neutron Science JCNS, Outstation at MLZ, Forschungszentrum Jülich GmbH); KOUTSIOUMPAS, Alexandros (JCNS); Dr MATTAUCH, Stefan (FZ-Juelich); STIERLE, Andreas (DESY / Universität Hamburg)*

The mobility of Fe in magnetite is a key ingredient towards a better understanding of its defect structure and resulting properties. For nanoparticles, which find a range of applications in medicine, spintronics, material science and catalysis, the near-surface is particularly important. Recent scanning tunnelling microscopy (STM) and low energy electron diffraction (LEED) studies of the  $(\sqrt{2} \times \sqrt{2})R45^\circ$  reconstructed (001) surface suggested a subsurface vacancy stabilisation model for this surface, later proved by surface x-ray diffraction (SXRD) [1,2]. Low energy electron microscopy (LEEM) experiments under catalytic conditions showed a regrowth process of Fe<sub>3</sub>O<sub>4</sub>-layers on (001) surfaces [3]. These results point towards an interesting interplay between cation vacancy formation and diffusion. We present the results of iron exchange at the interface between <sup>57</sup>Fe<sub>3</sub>O<sub>4</sub> thin-films and a Fe<sub>3</sub>O<sub>4</sub> (001) substrate after ultra high vacuum annealing at multiple temperatures. By exploiting the scattering length variation of <sup>57</sup>Fe and natural Fe, its interdiffusion across the film-substrate interface is characterized by neutron reflectometry at MARIA at MLZ [4]. The results on growth and diffusion are complemented by x-ray reflectometry data.

[1] Bliem, R. et al. Science. 346, 1215 (2014)

[2] Arndt, B. et al. Surf. Sci. 653, 76 (2016)

[3] Nie, S. et al., J. Am. Chem. Soc. 135, 10091 (2013)

[4] Schmidt, H. et al. Adv. Eng. Mat. 11, 446 (2009)



## In-situ X-ray diffraction during molecular beam epitaxy growth of III-V semiconductor nanowire structures

Monday, 12:15  
MW0001  
ID 383

**BAUMBACH, Tilo** (Karlsruhe Institute of Technology)

*FEIGL, Ludwig (Karlsruhe Institute of Technology); SCHROTH, Philipp (Karlsruhe Institute of Technology); JAKOB, Julian (Karlsruhe Institute of Technology); AL HUMAIDI, Mahmoud (University of Siegen); MOSTAFAVI KASHANI, Seyed Mohammad (University of Siegen); AL HASSAN, Ali (University of Siegen); VOGEL, Jonas (Johannes Gutenberg University of Mainz); DAVTYAN, Arman (University of Siegen); PIETSCH, Ullrich (University of Siegen)*

III-V semiconductor nanowires allow for a dislocation free integration of III-V semiconductors on Si substrates, which is highly interesting for optoelectronic applications. Ga-assisted GaAs nanowires exhibit a polytypism, which results in different segments of zincblende (ZB), its rotational twin (ZBT) and wurtzite (WZ) structure along the axial direction of nanowires. To better understand the growth processes, we monitor the shape and crystal structure of freestanding GaAs nanowires by in-situ X-ray diffraction (XRD), during fabrication and further processing by molecular beam epitaxy (MBE) growth on (111) plane Si substrates. We are using a portable MBE to do XRD studies at the in-house synchrotron at KIT and also at the large-scale synchrotrons PETRA III and ESRF. Employing X-ray focusing optics and patterned substrates, we are able to tune the number of nanowires we are probing from large ensembles down to a single nanowire. During experiments at P09 at PETRA III, we monitored the structural and radial evolution of the nanowires by repeatedly recording 3D reciprocal space maps around symmetric (111) and asymmetric Bragg reflections, comprising the (311) ZB, (220) ZBT and (10.3) WZ reflection [1] with a temporal resolution of 1-10min. By simultaneously performing reflective high-energy electron diffraction (RHEED) we gain additional sensitivity during the nucleation stage and to small changes during the growth process.

[1] Schroth et al. Nano Lett. 18, 101 (2018)



**Monday – September 17<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Micro symposium 1***

# ***In-situ and in-operando studies with special focus on energy materials and catalysis***

**MW1801**

*Each field is linked to the abstract's start page. Just click on the title!*

<p><b>11:00-11:30</b> <i>Helmut Ehrenberg</i> Electrochemical energy storage beyond lithium: mechanisms revealed by in operando synchrotron studies</p>
<p><b>11:30-11:45</b> <i>Günther Redhammer</i> Li-ion localisation and mobility in selected solid-state electrolytes probed by neutron diffraction</p>
<p><b>11:45-12:00</b> <i>P. Klaus Pranzas</i> Characterisation of hydrogen storage materials with photons and neutrons</p>
<p><b>12:00-12:15</b> <i>Werner Paulus</i> Phase diagram and redox behavior of (Nd/Pr)<sub>2</sub>NiO<sub>4+δ</sub> electrodes explored by in situ neutron powder and synchrotron single crystal diffraction during electrochemical oxygen intercalation</p>
<p><b>12:15-12:30</b> <i>Franziska Löhner</i> Understanding morphological degradation in organic photovoltaics with advanced scattering techniques</p>



## Electrochemical energy storage beyond lithium: mechanisms revealed by in operando synchrotron studies

Monday, 11:00  
MW1801  
ID 389

***EHRENBERG, Helmut (KIT)***

*FU, Qiang (KIT); DSOKE, Sonia (KIT); INDRIS, Sylvio (KIT); AZMI, Raheleh (KIT); KNAPP, Michael (KIT); TROUILLET, Vanessa (KIT)*

Electrochemical energy storage beyond lithium is of high relevance for a sustainable energy technology. However, qualitatively new concepts are needed for suitable electrodes, especially in the case of the intercalation of larger monovalent ions like Na<sup>+</sup> or K<sup>+</sup> or multivalent ions like Mg<sup>2+</sup>, Ca<sup>2+</sup> or Zn<sup>2+</sup>. One example for a promising Na-ion battery is presented based on symmetrical NASICON-structured Na<sub>2</sub>VTi(PO<sub>4</sub>)<sub>3</sub> electrodes [1]. The contribution of in situ synchrotron diffraction and X-ray absorption spectroscopy to unravel the underlying sodium storage mechanism and charge compensation behaviour is presented. Model systems for multivalent-ion insertion can also include hybrid batteries with two mobile metal ions in the electrolyte, where a metal like Mg is plated at the negative electrode, while Li- or Na-ions are inserted at the positive electrode [2,3]. This presentation summarizes some recent results on the underlying working mechanisms in such hybrid batteries as revealed by in operando diffraction using synchrotron radiation in combination with X-ray photoelectron spectroscopy (XPS).

[1] D. Wang, et al., Nat. Commun. 8 (2017) 15888.

[2] X. Bian, et al., Mater. Chem. A, 2017, 5, 600.

[3] Q. Fu, et al., Electrochemical and structural investigations of different polymorphs of TiO<sub>2</sub> in magnesium and hybrid lithium/magnesium batteries, Electrochim. Acta, subm.



## Li-ion localisation and mobility in selected solid-state electrolytes probed by neutron diffraction

Monday, 11:30  
MW1801  
ID 411

**REDHAMMER, Günther (Universität Salzburg)**

*RETTENWANDER, Daniel (TU Graz); BERENDTS, Stefan (TU Berlin); GANSCHOW, Steffen (Leibnitz-Institut für Kristallzüchtung); MEVEN, Martin (RWTH Aachen/ JCNS@MLZ); SENYSHYN, Anatoliy (MLZ)*

The search for new concepts and materials for energy related technology has become a demanding branch in materials sciences and one focus has been shifted to Li- and Na-ion batteries for ready storage and use of energy. One bottle-neck is the use of liquid electrolytes, which induce a number of limitations, device failure due to corrosion and dendritic intergrowth between cathode and anode, etc. Thus, suitable (solid state) electrolyte materials are: among others, two different material groups have been identified, the first ones are Li-bearing garnet type LLZOs, a second are the NASICON-type compounds. A great advantage of NASICON-type materials is, that they work both as Li-ion as well as Na-ion conducting compounds. Suitable solids for the use in batteries are characterized by a high mobility of alkaline ions. This induces several challenges in e.g. localizing light elements, which might be an issue at all using conventional techniques. Neutron diffraction (ND) at non-ambient conditions /temperatures) is the experimental method of choice to give a deep insight into light element distribution in the crystal structure, ion-mobility and transport properties in modern energy related materials. In the talk we will report on recent progress obtained using single crystal and powder neutron diffraction of doped LLZO garnet type  $\text{Li}_7\text{La}_3\text{ZrO}_{12}$ , Libased NASCON type  $\text{Li}_{1+x}\text{Ti}_{2-x}(\text{Al,Fe})(\text{PO}_4)_3$  and anti-perovskite-type  $\text{Li}_{3-x}(\text{OH})_x\text{Cl}$  materials and highlight the fancy possibilities of ND.



## Characterisation of hydrogen storage materials with photons and neutrons

Monday, 11:45  
MW1801  
ID 65

**PRANZAS, P. Klaus (HZG)**

*BÖRRIES, Stefan (HZG); KARIMI, Fahim (HZG); PISTIDDA, Claudio (HZG); HOELL, Armin (HZB); DORNHEIM, Martin (HZG); KLASSEN, Thomas (HZG); SCHREYER, Andreas (ESS)*

Hydrogen is a promising energy carrier for the future, especially for mobile applications. It can be stored safely and reversibly at high volumetric densities in hydrogen storage tanks filled with light metal hydrides. Reactive Hydride Composites (RHCs) are metal hydride mixtures that are very promising hydrogen storage materials due to high hydrogen densities, stability and safety. The hydrogen sorption kinetics of the RHCs is distinctly improved by high-energy ball milling and the addition of suitable additives. Phase transformations and changes in the nanostructure were characterised using in situ synchrotron radiation-powder X-ray diffraction (SR-PXD), X-ray absorption spectroscopy (XAS), anomalous small-angle X-ray scattering (ASAXS) and small-angle neutron scattering (SANS) in order to get a deeper insight into the complex hydrogen sorption processes in the different RHC systems. In situ Neutron Radiography (NR) experiments were performed for time-resolved investigations of the hydrogenation process of metal hydride powder beds and pellets inside a hydrogen storage tank. Neutron Computerized Tomography (NCT) provided additional 3D information about material structure and quantitative hydrogen distribution and allowed correlation studies of the effect of temperature field and material packing density. The results give important information for tailoring of the hydrogen storage materials and tank systems in terms of capacity, kinetics and safety.

## Phase diagram and redox behavior of (Nd/Pr)<sub>2</sub>NiO<sub>4+δ</sub> electrodes explored by in situ neutron powder and synchrotron single crystal diffraction during electrochemical oxygen intercalation

Monday, 12:00  
MW1801  
ID 417

**PAULUS, Werner (Institut Charles Gerhardt Montpellier, UMR 5253 CNRS-UM-ENSCM)**

*WAHYUDI, Olivia (Institut Charles Gerhardt Montpellier, UMR 5253 CNRS-UM-ENSCM, Université de Montpellier, France); CERETTI, Monica (Institut Charles Gerhardt Montpellier, UMR 5253 CNRS-UM-ENSCM, ); MAITY, Avishek (Institute for Physical Chemistry, Georg August University Göttingen, Outstation at Heinz Maier-Leibnitz Zentrum ); DUTTA, Rajesh (Institut Charles Gerhardt Montpellier, UMR 5253 CNRS-UM-ENSCM); MEVEN, Martin (Institute of Crystallography, RWTH Aachen University and JCNS@MLZ); CHERNYSHOV, Dmitry (Swiss Norwegian Beamlines SNBL@ESRF)*

Oxygen intercalation/deintercalation in Pr<sub>2</sub>NiO<sub>4+δ</sub> and Nd<sub>2</sub>NiO<sub>4+δ</sub> was followed by in situ neutron powder and single crystal synchrotron diffraction during electrochemical oxidation/reduction, in dedicated reaction cells [1]. For both systems three phases, all showing the same line-width, were identified. The starting phases, Pr<sub>2</sub>NiO<sub>4.23</sub> and Nd<sub>2</sub>NiO<sub>4.24</sub>, considered with an average orthorhombic Fmmm symmetry, although both show a slight monoclinic distortion, get reduced in a 2-phase reaction step to tetragonal intermediate phases with  $0.07 \leq \delta \leq 0.10$  and P42/ncm space group, which on further reduction transform, again in a 2-phase reaction step, towards the respective stoichiometric (Pr/Nd)<sub>2</sub>NiO<sub>4.0</sub> phases, with Bmab space group. Electrochemical oxidation does, however, not proceed fully reversibly for both cases: while the re-oxidation of Nd<sub>2</sub>NiO<sub>4+δ</sub> is limited to the tetragonal intermediate phase with  $\delta = 0.10$ , the homologous Pr<sub>2</sub>NiO<sub>4+δ</sub> can be re-oxidized up to  $\delta = 0.17$ , showing orthorhombic symmetry. For the intermediate tetragonal phase, we were able to establish for Pr<sub>2</sub>NiO<sub>4.09</sub> complex anharmonic displacement behavior for the Pr<sub>2</sub>O<sub>2</sub> rock salt layer, as analyzed by single crystal neutron diffraction and Maximum Entropy Analysis, in agreement with a low-T diffusion pathway for oxygen ions, activated by low energy phonon modes [2-4].

[1] M. Ceretti, O. Wahyudi, G. André, M. Meven, A. Villesuzanne, W. Paulus, Inorg. Chem. 57,8, 4656-66 (2018)

[2] M. Ceretti, O. Wahyudi, A. Cousson, A. Villesuzanne, M. Meven, B. Pedersen, J. M. Bassat, W. Paulus, J. Mat. Chem. A, 3,42 (2015), 21140-21148

[3] A. Piovano, A. Perrichon, M. Boehm, M. R. Johnson, W. Paulus, Phys. Chem. Chem. Phys., 18 (2016) 17398-17403

[4] Paulus, H. Schober, S. Eibl, M. Johnson, T. Berthier, O. Hernandez, M. Ceretti, M. Plazanet, K. Conder, C. Lamberti, J. Am. Chem. Soc. 130 (47) (2008) 16080-85



## Understanding morphological degradation in organic photovoltaics with advanced scattering techniques

Monday, 12:15  
MW1801  
ID 448

**LÖHRER, Franziska (Lehrstuhl für Funktionelle Materialien, Department für Physik E13, TU München)**

*YANG, Dan (TU München, Physik-Department, LS Funktionelle Materialien); SCHAFFER, Christoph (TUM - Physics Dept. - Functional Materials); WANG, Weijia (TUM); MOSEGUÍ GONZÁLEZ, Daniel (TU München); BERNSTORFF, Sigrid (Elettra-Sincrotrone Trieste); ROTH, Stephan (DESY/KTH); MÜLLER-BUSCHBAUM, Peter (TU München, Physik-Department, LS Funktionelle Materialien)*

Organic photovoltaics (OPV) have received high attention in recent years as an interesting alternative to conventional solar cells. Recent research efforts focus on enhancing the photovoltaic performance in order to make organic solar cells feasible for industrial purposes. This has led to the development of low band-gap materials with reported power conversion efficiencies surpassing the magic limit of 10 %. [1] However, especially high-efficiency polymers are sensitive to various degradation processes, which strongly decrease their lifetime in comparison to commercially available inorganic photovoltaics. Several issues concerning the optimal thin film morphology and architecture have to be addressed to make organic solar cells a potential candidate for mass market applications. The work of our group takes a deeper look at the morphologically induced aging processes of photoactive materials during operation of OPV devices, using in-operando GISAXS and GIWAXS. [2-5] Following the photovoltaic and structural properties of the devices simultaneously with advanced scattering techniques gives valuable insights into the degradation pathways inside the photoactive organic layer.

[1] Zhao, et al., Nat. Energy 1 (2016)

[2] Schaffer, et al., Adv. Mater. 25, 46 (2013)

[3] Wang, et al., J. Mater. Chem. A 3 (2015)

[4] Schaffer, et al., Adv. Energy Mater. 6, 9 (2016)

[5] Moseguí González, et al., ACS Appl. Mater. Interfaces 9, 4, (2017)



**Monday – September 17<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Micro symposium 2***

# ***Structural biology: Instrumentation and methods, current trends and topics***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:00-11:15**

*Christoph Mueller-Dieckmann*

Facilities for Structural Biology at the ESRF: present and future

**11:15-11:30**

*Manfred Rößle*

Investigations on Structure and Dynamics of Proteins by SAXS

**11:30-11:45**

*Sebastian Guenther*

P11 at PETRA III: A Versatile Beamline for Serial and High-Throughput Crystallography

**11:45-12:00**

*Thomas Schneider*

EMBL beamlines for macromolecular crystallography at PETRA III

**12:00-12:15**

*Martin Gerlach*

Facilities for Macromolecular Crystallography at the HZB

**12:15-12:30**

Discussions



### Facilities for Structural Biology at the ESRF: present and future

Monday, 11:00  
MW0350  
ID 54

*MUELLER-DIECKMANN, Christoph*

A major success story of the European Synchrotron Radiation Facility (ESRF, the European Synchrotron) has been its facilities for Structural Biology. Here, end-stations for experiments in Macromolecular Crystallography, BioSAXS and in crystallo spectroscopy have recently been supplemented by the installation of a high-end cryo-electron microscope. The very high level of productivity of these end-stations has been driven both by the scientific excellence of the ESRF's Structural Biology user community and by a very high level of beamline automation which allows experimenters to get the best out of even the most difficult of samples. This talk will provide an overview of the automation currently available, including a completely "hands off" beamline and protocols for the construction of useful, complete diffraction data sets from multi-crystal data collection experiments. December 2018 will see the ESRF temporarily close for the construction of the Extremely Bright Source (EBS) which will have a horizontal emittance of the electron beam in the storage ring ~ 30 times smaller than is currently the case. The proportionate reduction in X-ray beam sizes and divergences will allow the construction of new generation ESRF MX beamlines with flux densities at the sample position up to 5 orders of magnitude higher than at present. This talk will thus also look forward to the possibilities that the ESRF-EBS will afford in the study of structural biology.



## **Investigations on Structure and Dynamics of Proteins by SAXS**

**Monday, 11:18  
MW0350  
ID 345**

***RÖBLE, Manfred (University of Applied Science Luebeck)***

*SCHEWA, Siawosch; ZICKMANTEL, Till*

Modern X-ray sources such as synchrotrons and free-electron lasers provide the possibility of ultra-fast data recording within micro-seconds. Small angle X-ray scattering (SAXS) on biological macromolecules is one tool for exploring the kinetic and dynamic of structural alteration of proteins during the working cycle. Advanced sample environments based on microfluidic devices allows handling sample volumes of several pico-liters. For instance fast mixing of liquids is used for time resolved scattering experiments. While such techniques allow analyzing the kinetics of chemical or biochemical reactions, need investigations on the dynamics of a system a more sophisticated approach.

Classical pump-probe experiments facilitates the analysis of reaction dynamics. For these investigations, for instance an ultra-short laser pulse is triggering the reaction in the sample. The high flux synchrotron beam is used for investigated the structural response of the system. Proteins are expected to exhibit functionally relevant, global domain and sub domain collective modes in the terahertz (THz) frequency range. These normal modes can be triggered by THz radiation. The redistribution of vibrational modes translates into movements and rearrangement of large protein domains in the range up to nm. Such experiments need, beside the brilliance of the X-ray beam suitable fast detector system for recording the data in short time frames.



## P11 at PETRA III: A Versatile Beamline for Serial and High-Throughput Crystallography

Monday, 11:36  
MW0350  
ID 119

**GUENTHER, Sebastian (Deutsches Elektronen-Synchrotron DESY)**

*BURKHARDT, Anja (Deutsches Elektronen-Synchrotron DESY); LORBEER, Olga (Deutsches Elektronen-Synchrotron DESY); CROSAS, Eva (Deutsches Elektronen-Synchrotron DESY); PAKENDORF, Tim (Deutsches Elektronen-Synchrotron DESY); REIME, Bernd (Deutsches Elektronen-Synchrotron DESY); MEYER, Jan (Deutsches Elektronen-Synchrotron DESY); FISCHER, Pontus (Deutsches Elektronen-Synchrotron DESY); STUEBE, Nicolas (Deutsches Elektronen-Synchrotron DESY); WARMER, Martin (Deutsches Elektronen-Synchrotron DESY); MEENTS, Alke (Deutsches Elektronen-Synchrotron DESY)*

P11 at PETRA III in Hamburg is dedicated to structural investigations of biological samples at different length scales. The beamline provides two state-of-the-art endstations: a crystallography experiment which is in user operation since 2013 [1] and an X-ray microscope which is utilizing tender X-rays between 2.4 and 10 keV and is currently under construction [2]. In future, a third experimental endstation for serial crystallography and pump-probe experiments will be available. Basis of beamline design was to make full use of the brilliant source properties of the PETRA III synchrotron and to deliver most of the photons from the source into a very small focal spot at the sample position.

The P11 optics concept involves the generation of a secondary source at 65.5 m using three dynamically bendable KB mirrors. A second KB system which is installed further downstream can be used for refocusing the X-ray beam down to  $4 \times 9 \mu\text{m}^2$  ( $v \times h$ ) with full flux from the source ( $1.3 \times 10$  ph/s at 12 keV). Smaller beam sizes down to  $1 \times 1 \mu\text{m}^2$  with more than  $2 \times 10^{11}$  ph/s in the focus can be obtained by slitting down the secondary source at the cost of flux.

The flexible X-ray optics allow for tailoring the beam properties to the needs of the experiment: A large parallel beam is available for structure determinations from large unit cell systems, such as large molecular complexes [3]. A highly intense microbeam allows for serial crystallography experiments on microcrystals using liquid delivery systems like jets [4] and the newly developed tape-drive system [5] or micro-patterned silicon chips for fixed targets [6, 7].

The P11 crystallography experiment is operated between 5.5 and 30 keV and provides full SAD/MAD capability. The endstation is equipped with a high precision single axis goniostat with a combined sphere of confusion of less than 100 nm. Crystals can be rapidly exchanged in less than 20 s using a sample changing robot which is equipped with an inhouse-designed sample gripper. In addition, a large capacity storage Dewar provides space for 23 uni-pucks (368 samples). The Pilatus 6M-F detector in place allows for fast data collection with frame rates of up to 25 Hz. A full data set can be typically collected in less than 2 min. These design parameters make P11 ideally suited for screening a large number of crystals in very short time.

[1] A. Burkhardt et al., EPJ Plus 131:56 (2016)

[2] A. Meents et al., Proc. SPIE 8851, 88510K (2014)

[3] K. Schulte et al., BMC Biology 14:14 (2016)

[4] F. Stellato et al., IUCrJ 1, 204 (2014)

[5] K. R. Beyerlein, IUCrJ 4, 439 (2017)

[6] P. Roedig et al., Sci. Rep. 5, 10451 (2015)

[7] P. Roedig et al., J. Appl. Cryst. 49, 968 (2016)

## EMBL beamlines for macromolecular crystallography at PETRA III

Monday, 11:54  
MW0350  
ID 130

**SCHNEIDER, Thomas (EMBL c/o DESY)**

*BOURENKOV, Gleb (EMBL c/o DESY); POMPIDOR, Guillaume (EMBL c/o DESY); HAKANPÄÄ, Johanna; BENTO, Isabel; PANNEERSELVAM, Saravanan (EMBL c/o DESY); VON STETTEN, David (EMBL c/o DESY); AGTHE, Michael (Univ. Hamburg); HORRELL, Sam (Univ. Hamburg); PEARSON, Arwen (Univ. Hamburg); FIEDLER, Stefan (EMBL c/o DESY)*

EMBL-Hamburg operates two beamlines for macromolecular crystallography, P13 and P14, at PETRA III (DESY, Hamburg).

P13 delivers high photon fluxes at energies down to 4 keV. Combining X-rays in the 4-6 keV energy range with beam sizes down to 15  $\mu\text{m}$  diameter while maintaining high photon flux and using standard mounting systems (SPINE pins) and robotics allows to solve the crystallographic phase problem via S-SAD phasing; recently the feasibility of MAD-phasing at the Ca K-edge (4.05 keV, 3.1 Å) has been demonstrated P14 can be run in two modes, one providing a collimated homogeneous beam that can be shaped to any size between 10 and 200  $\mu\text{m}$ , the second mode producing micro-focus conditions with a beam size on the 5  $\mu\text{m}$  scale. Under micro-focus conditions, serial data collections - both under cryogenic and in situ conditions - employing 'serial helical scans'-strategies can be executed conveniently from the MXCuBE user interface.

While several pump-probe and time-resolved experiments have been successfully performed on P14, we are presently constructing a second endstation on P14 to provide flexible beam and sample conditions for pump-probe experiments (in collaboration with the University of Hamburg). The beamlines are embedded in the Integrated Facility for Structural Biology that offers access to up-stream service such as characterization of samples prior to crystallization, high throughput crystallization, and automatic crystal harvesting with a CrystalDirect™ Harvester.



## Facilities for Macromolecular Crystallography at the HZB

Monday, 12:12  
MW0350  
ID 141

**GERLACH, Martin (Helmholtz-Zentrum Berlin)**

*FEILER, Christian (Helmholtz-Zentrum Berlin); FÖRSTER, Ronald (Helmholtz-Zentrum Berlin); GLESS, Christine (Helmholtz-Zentrum Berlin); HAUß, Thomas (Helmholtz-Zentrum Berlin); HELLMIG, Michael (Helmholtz-Zentrum Berlin); KASTNER, Alexandra (Helmholtz-Zentrum Berlin); SCHMUCKERMAIER, Lukas (Helmholtz-Zentrum Berlin); STEFFIEN, Michael (Helmholtz-Zentrum Berlin); TABERMAN, Helena (Helmholtz-Zentrum Berlin); WILK, Piotr (Helmholtz-Zentrum Berlin); WOLLENHAUPT, Jan (Helmholtz-Zentrum Berlin); WEISS, Manfred S. (Helmholtz-Zentrum Berlin)*

The Helmholtz-Zentrum Berlin (HZB) operates three beamlines for macromolecular crystallography (MX) at the electron storage ring BESSY II [1,2]. BL14.1 and BL14.2 are tunable in the photon energy range from 5 to 16 keV, while BL14.3 is a fixed-energy side station (13.8 keV). They feature state-of-the-art experimental stations and ancillary facilities, serving more than 100 research groups across Europe. More than 2500 protein structures measured at BESSY II have resulted in protein data base (PDB) depositions so far, and with more than 600 PDB depositions in 2016, they are currently among the five most productive MX-stations in the world. The experimental endstation of BL14.1 provides high degree of automation and is equipped with a Pilatus 6M detector, a CATS sample changer robot and an MD2 multi-axis goniometer. Recently BL14.2 underwent a comprehensive upgrade to increase the performance in terms of sample throughput, allowing for large automated fragment-screening campaigns in the near future. It features a Pilatus3S 2M detector, a G-Rob sample changer and a piezo-controlled nanodiffractometer. Its sample dewar can accommodate up to 294 samples, supporting both, SPINE- and UNIPUCK standards. All standard data collection procedures, such as SAD and MAD, are possible, as well as longwavelength measurements and element identification using an X-ray fluorescence detector. Further experimental possibilities are radiation induced phasing experiments using a pulsed UV-laser and in situ crystal screening, supporting several types of 96-well plates. An HC-Lab dehydration device allows for controlled crystal dehydration, and in combination with a rapid nozzle exchanger a cryojet can be used to freeze samples in a defined hydration state within a fraction of a second [3]. Furthermore, the HZB-MX group operates an S1 BioLab which supports the complete workflow from protein purification to crystallization.

[1] U. Mueller et al. (2012), J. Synch. Rad. 19, 442

[2] U. Mueller et al. (2015), Eur. Phys. J. Plus 130, 141

[3] M. W. Bowler et al. (2015), Cryst. Growth Des. 15, 1043



**Monday – September 17<sup>th</sup>, 2018**

**14:30-16:00**

## ***Parallel session 2***

### ***Soft matter***

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

**14:30-14:45**

*Stephan Förster*  
Soft quasicrystals

**14:45-15:00**

*Matthias Karg*  
Soft photonic crystals from plasmonic core/shell particles: A spectroscopy and SANS study

**15:00-15:15**

*Aurel Radulescu*  
Contrast-Variation Small-Angle Neutron Scattering – A Powerful Method to Unravel the Micro-Structure in Polymer Electrolyte Membranes

**15:15-15:30**

*Fajun Zhang*  
Dynamic Arrest of Liquid-Liquid Phase Separation in Protein Solutions Studied Using Ultra-Small Angle X-ray Scattering (USAXS)

**15:30-15:45**

*Oliver Wrede*  
Swelling kinetics of N-n-propylacrylamide based microgels studied by time-resolved Small Angle Neutron Scattering

**15:45-16:00**

*Mareike Töpperwien*  
3D virtual histology of neuronal tissue by propagation-based x-ray phase-contrast tomography



## Soft quasicrystals

Monday, 14:30  
MW2001  
ID 286

*FÖRSTER, Stephan (Forschungszentrum Jülich)*

*DULLE, Martin (Forschungszentrum Jülich); JURCZYK, Tobias (Forschungszentrum Jülich); FRIELINGHAUS, Henrich (Forschungszentrum Jülich)*

Quasicrystals have a unique aperiodic order, which leads to diffraction patterns of 8-, 10-, or 12- fold rotational symmetry. They are usually found for binary and ternary metal alloys. Beginning in 2004 there have been reports on quasicrystalline structures formed by soft materials such as dendrimers, polymers and surfactants. This indicates that there might be very general principles that favor the formation of quasicrystals, and if this would be the case, they could be rationally designed with characteristic length scales up to the submicron range for photonic applications. We have discovered quasicrystalline phases with 12- and 18-fold rotational symmetry for PI-PEO block copolymer polymer micelles in water, and for PS-PI block copolymer micelles in diethylphthalate. Their stability range and phase transitions into FCC, BCC, Frank-Kasper and disordered phases as a function of concentration, temperature and molecular weight can be well determined by rheo-SAXS and rheo-SANS experiments. Using MD-simulations we are able to map the stability regions of a large variety of crystalline (FCC, BCC, HCP, Cp4, BC8, hP2, hP10), quasi-crystalline (IdQ12, hdQ12, IQC) and Frank-Kasper phases (A15, H) in terms of the micellar volume fraction and core/shell size ratio. The phase diagram for the first time links previous reports of quasicrystals in nanoparticles, polymers and MD-simulations to provide a fundamental understanding of the structure and stability of soft quasicrystals.



## Soft photonic crystals from plasmonic core/shell particles: A spectroscopy and SANS study

Monday, 14:45  
MW2001  
ID 303

**KARG, Matthias (Physical Chemistry I, Heinrich-Heine-University)**

*RAUH, Astrid (Physical Chemistry I, University of Bayreuth); CARL, Nico O. (Large Scale Structures Group, Institut Laue-Langevin); SCHWEINS, Ralf (Large Scale Structures Group, Institut Laue-Langevin)*

Photonic crystals are well known for their fascinating angle-dependent opalescence. In contrast to hard sphere building blocks, crystals from soft spheres such as microgels allow for external actuation, e.g. melting and recrystallization induced by pH or temperature changes.[1] In addition, soft spheres can be deformed and thus packing fractions well above the hard sphere limit are accessible. A drawback with respect to the optical diffraction of soft photonic crystals is the typically low refractive index modulation inside the material. This asks for pathways to increase the refractive index contrast in the periodic superstructure. We prepared cm-sized soft photonic crystals from core/shell microgels with absorbing nanocrystal cores (gold nanoparticles) and systematically studied the crystal structure and diffractive properties.[ 2] As non-absorbing references, we prepared pitted particles from the same microgel batches by dissolution of the nanocrystal cores. The resulting purely organic microgels allowed for a direct comparison of structural and optical properties. Dynamic and static light scattering as well as small angle neutron scattering (SANS) were used to study the core/shell and pitted particles in the dilute state on different length scales. The crystal structure of samples at high volume fractions was analysed by SANS. Independent on the presence or absence of the gold cores, we found fcc crystal structures with pronounced Bragg peaks of several orders. We identify the role of small absorbing nanocrystal cores in soft photonic crystals and show options to alter the photonic behavior of such crystals in future studies.

[1] M. Karg, T. Hellweg, P. Mulvaney, Adv. Funct. Mater. 21 (2011), 468.

[2] A. Rauh, N. Carl, R. Schweins, M. Karg, Langmuir 34 (2018), 854.



## Contrast-Variation Small-Angle Neutron Scattering – A Powerful Method to Unravel the Micro-Structure in Polymer Electrolyte Membranes

Monday, 15:00  
MW2001  
ID 355

*RADULESCU, Aurel (Forschungszentrum Jülich GmbH)*

Specially designed polymer electrolyte membranes for fuel cells (PEMFC) applications must present special properties such as high ionic conductivity, adequate mechanical strength, chemical and electrochemical stability under operating conditions, moisture control in stack, and low cost production. Currently, the most used material for such applications is the Nafion, which, despite its excellent properties, shows some disadvantages such as the high cost and the need for special safety and supporting equipment during its manufacture and use. This has prompted the research towards alternative polymers. On the other hand, the properties of PEMFCs derive from the microphase separation of a hydrophilic ionic material from a hydrophobic substance. Therefore, for newly designed PEMFCs the understanding of the micro-structure, such as crystalline domains, conducting regions, as well as distribution of ionic groups and water in conducting regions is essential for optimizing the relationship between structure and properties. Through the manipulation of the scattering contrast between different regions and components of such complex systems the small-angle neutron scattering (SANS) enables a detailed and unique micro-structural characterization under functional conditions of temperature and humidity. In this communication the principle and representative examples of experimental investigations carried out on new PEMFCs at the KWS-2 SANS instrument of JCNS at MLZ will be reported.



## Dynamic Arrest of Liquid-Liquid Phase Separation in Protein Solutions Studied Using Ultra-Small Angle X-ray Scattering (USAXS)

Monday, 15:15  
MW2001  
ID 17

**ZHANG, Fajun** (*University of Tuebingen*)

*DA VELA, Stefano (Institut für Angewandte Physik, Universität Tübingen); BRAUN, Michal K.; MÖLLER, Johannes; SCHREIBER, Frank*

The interplay between liquid-liquid phase separation (LLPS) and glass formation leads to a dynamically arrested state in colloidal and protein systems. However, many details regarding the transition from LLPS to the arrested state and their responses to the subtle changes of the quench depth are still not fully understood. Further more, the interplay between glass formation and LLPS, i.e. whether the glass line follows the equilibrium binodal or enters into the coexistence region, is still a matter of debate. Here we have employed the USAXS technique to study the kinetics of LLPS and arrested state in protein systems with a high temporal and spatial resolution. The two protein systems studied were bovine gamma-globulin in the presence of PEG, featuring an upper critical solution temperature (UCST) phase behavior, and BSA with YCI3, featuring a lower critical solution temperature (LCST) phase behavior. For both systems, the time evolution of the characteristic length during phase separation was followed by USAXS over a broad range of time scales. Depending on the quench depths, classical coarsening kinetics as well as fully arrested state were identified. The transition between these two states was linked with a temporarily arrested state with three-step growth kinetics. Further more, the scattering invariant were used to determine the glass line in the arrested state which provides new insights for the interplay between glass formation and LLPS in various protein systems.



## Swelling kinetics of N-n-propylacrylamide based microgels studied by time-resolved Small Angle Neutron Scattering

Monday 15:30  
MW2001  
ID 379

**WREDE, Oliver (Bielefeld University)**

*REIMANN, Yvonne (University of Cologne); LÜLSDORF, Stefan (University of Stuttgart); EMMRICH, Daniel (Bielefeld University); SCHNEIDER, Kristina (University of Stuttgart); SCHMID, Andreas Josef (Physikalische und Biophysikalische Chemie, Universität Bielefeld); ZAUSER, Diana (University of Stuttgart); HERTLE, Yvonne (Universität Bielefeld, PCIII); BEYER, André (Bielefeld University); SCHWEINS, Ralf (Institut Laue-Langevin); GÖLZHÄUSER, Armin (Bielefeld University); HELLWEG, Thomas (Universität Bielefeld, PCIII); SOTTMANN, Thomas (University of Stuttgart)*

Temperature responsive microgels are particles in the size range between 100 nm and 1  $\mu\text{m}$  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 by time-resolved small angle neutron scattering (SANS). Preceding photon correlation spectroscopy (PCS) 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 [1], 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. While the swelling occurred on a time scale of around 1 ms the deswelling was much slower ( $\sim 10$  ms). Together with static SANS and PCS spectroscopy we could show that the differences in the phase transition dynamics can be associated the underlying complex particle architecture, which exhibits a core region with a high density surrounded by a loosely connected shell.

[1] B. Wedel, Y. Hertle, O. Wrede, J. Bookhold, T. Hellweg, *Polymers*, 2016, 8, 162.



## 3D virtual histology of neuronal tissue by propagation-based x-ray phase-contrast tomography

Monday, 15:45  
MW2001  
ID 425

**TÖPPERWIEN, Mareike** (*Institute for X-Ray Physics, University of Göttingen*)

*STADELMANN, Christine* (*Institute for Neuropathology, University Medical Center Göttingen*); *SALDITT, Tim* (*Institute for X-Ray Physics, University of Göttingen*)

Studies of the brain cytoarchitecture in mammals are routinely performed by histology, i.e. by examining the tissue under a light microscope after serial sectioning and subsequent staining. The procedure is labor-intensive and prone to artifacts due to the slicing procedure. While it provides excellent results on the twodimensional (2d) slices, the 3d anatomy can only be determined after aligning the individual sections, leading to a non-isotropic resolution within the tissue. X-ray tomography offers a promising alternative due to the potential resolution and high penetration depth which enables non-destructive imaging of the sample's density distribution at high detail. In order to visualize weakly absorbing soft tissue samples, the phase-shift induced in the (partially) coherent beam is used for contrast formation via free space propagation between the sample and the detector.

Experiments were carried out at our waveguide-based holo-tomography instrument GINIX at DESY. This setup allows for high resolution recordings with adjustable field of view and resolution. We optimize for contrast and resolution by comparing different preparation techniques, reaching sub-cellular resolution in mm-sized neuronal tissue from human and mouse. Subsequent automatic cell segmentation provides access to the 3d cellular distribution within the tissue, enabling the quantification of the cellular arrangement and allowing for extensive statistical analysis based on several thousands of cells.



**Monday – September 17<sup>th</sup>, 2018**  
**14:30-16:00**

## ***Parallel session 5***

### ***Thin films, 2D and surfaces***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>14:30-14:45</b> <i>S. Stankov</i> Lattice dynamics of ultrathin EuO films and interfaces
<b>14:45-15:00</b> <i>Alexander Hinderhofer</i> Crystallization Dynamics of Perovskite Thin Films Studied by Real-time Grazing Incidence Diffraction
<b>15:00-15:15</b> <i>David Keeble</i> Point defect identification in perovskite oxide thin films and surfaces using a variable energy high intensity positrons
<b>15:15-15:30</b> <i>Moritz Hoesch</i> Three-dimensional Fermi surfaces from Diamond Light Source
<b>15:30-15:45</b> <i>Francesco Allegretti</i> Application of the X-ray Standing Wave Technique to Materials Research
<b>15:45-16:00</b> <i>Markus Trunk</i> Concentration profiles in thin films obtained from Neutron Depth Profiling at the PGAA facility



## Lattice dynamics of ultrathin EuO films and interfaces

Monday, 14:00  
MW0001  
ID 384

**STANKOV, S. (Karlsruhe Institute of Technology, Karlsruhe, Germany)**

*PRADIP, R. (Karlsruhe Institute of Technology, Karlsruhe, Germany); PIEKARZ, P. (Polish Academy of Sciences, Krakow, Poland); MERKEL, D. G. (Hungarian Academy of Sciences, Budapest, Hungary); KALT, J. (Karlsruhe Institute of Technology, Karlsruhe, Germany); WALLER, O. (Karlsruhe Institute of Technology, Karlsruhe, Germany); CHUMAKOV, A. I. (European Synchrotron Radiation Facility, Grenoble, France); RÜFFER, R. (European Synchrotron Radiation Facility, Grenoble, France); OLES, A. M. (Jagiellonian University, Krakow, Poland); PARLINSKI, K. (Polish Academy of Sciences, Krakow, Poland); BAUMBACH, T. (Karlsruhe Institute of Technology, Karlsruhe, Germany); STANKOV, S. (Karlsruhe Institute of Technology, Karlsruhe, Germany)*

Europium monoxide (EuO) is the first rare-earth semiconducting oxide known for its giant magneto-optic Kerr [1] and Faraday [2] effects, metal-insulator transition [3] and anomalous Hall effect [4]. Presently, it is one of the favored candidates for applications as a spin filter in future spintronic devices due to the large exchange splitting of its conduction band [5]. Employing inelastic X-ray scattering, nuclear inelastic scattering and first-principles theory we determined the lattice dynamics of this material and discovered a giant and anisotropic spin-phonon coupling [6]. This imposed the intriguing question about the manifestation of this phenomenon in thin and ultrathin films related to the proposed applications. Using in situ nuclear inelastic scattering on  $^{151}\text{Eu}$  we investigated the phonon density of states of EuO films with thickness between 8 nm and of 1 atomic layer. The experimental results unveiled drastic lattice dynamics modifications in the ultrathin EuO films that can be comprehensively understood by the help of first-principles theory. This work was financially supported by the Helmholtz Association (VHNG-625) and BMBF (05K16VK4).

[1] J. H. Greiner and G. J. Fan, Appl. Phys. Lett. 9, 27 (1966)

[2] K. Y. Ahn, J. Appl. Phys. 41, 1260 (1970)

[3] Y. Shapira, S. Foner, and R. Aggarwal, Phys. Rev. B 8, 2316 (1973)

[4] Y. Shapira, S. Foner, and R. Aggarwal, Phys. Rev. B 8, 2316 (1973)

[5] P. G. Steeneken et al., Phys. Rev. Lett. 88, 047201 (2002)

[6] R. Pradip et al., Phys. Rev. Lett. 116, 185501 (2016)



## Crystallization Dynamics of Perovskite Thin Films Studied by Real-time Grazing Incidence Diffraction

Monday, 14:45  
MW0001  
ID 257

*HINDERHOFER, Alexander (University of Tübingen)*

*SCHREIBER, Frank (University of Tübingen); GRECO, Alessandro (University of Tübingen)*

New materials for thin film applications, like photovoltaics and light emitting diodes, are often deposited from solution. One of these relatively new material classes is hybrid organic-halide perovskite. The performance of such perovskite thin films depends not only on their composition, but also their structural and morphological features.[1] We applied in situ real-time grazing incidence wide angle X-ray scattering (GIWAXS) to determine the reaction rate, domain size and preferred orientation in several perovskite thin films during crystallization from solution. The relevant time scales for the crystallization range from the subsecond regime up to several minutes. The perovskite thin films are prepared with the so-called two-step conversion method, which involves the deposition of an iso-propanol solution onto a lead halide precursor ( $\text{PbX}_2$ ,  $X=\text{I}, \text{Br}, \text{Cl}$ ) via drop casting. The solution contains a defined amount of either methylammonium halide ( $\text{CH}_3\text{NH}_3\text{X}$ ) or formamidinium halide ( $\text{CH}(\text{NH}_2)_2\text{X}$ ). We will discuss how different lead halide precursors in combination with different organic halide solutions can impact the quality and time scale of the structural and morphological development of the perovskite thin films.

[1] N. Arora et al. Science, 358, 768 (2017).



## Point defect identification in perovskite oxide thin films and surfaces using a variable energy high intensity positrons

Monday, 15:00  
MW0001  
ID 357

*KEEBLE, David (University of Dundee)*

*EGGER, Werner (Universität der Bundeswehr München); HENSLING, Felix (Forschungszentrum Jülich); GUNDEL, Felix (RWTH Aachen University); DITTMANN, Regina (Forschungszentrum Jülich)*

Variable energy positron annihilation lifetime spectroscopy measurements using the high intensity positron beam have been performed on high quality SrTiO<sub>3</sub>, and SrTiO<sub>3</sub>/LaAlO<sub>3</sub>, thin films grown by pulsed laser deposition (PLD) and on near surface region of SrTiO<sub>3</sub> substrates subjected to PLD growth conditions. The measurements were performed using the NEPOMUC beamline at Heinz Maier-Leibnitz Zentrum (MLZ), Garching. The presence of vacancy-related point defects is detected, at concentrations below approximately 100 ppm. The positron lifetime spectra are deconvolved and the lifetime component characteristic of the two types of perovskite oxide, ABO<sub>3</sub>, cation monovacancy, the B-site and A-site vacancies, are identified and are consistent with density functional theory calculated positron lifetime values. Measurements on the process modified near surface region of the SrTiO<sub>3</sub> substrate provide evidence for the presence of Ti-vacancy O-vacancy divacancy complexes. Changes in positron trapping with UV illumination are observed. The measurements also observe changes in the vacancy defect content on annealing SrTiO<sub>3</sub>/LaAlO<sub>3</sub> structures, the conductivity of the interface is also changed.



## Three-dimensional Fermi surfaces from Diamond Light Source

Monday, 15:15  
MW0001  
ID 189

**HOESCH, Moritz (DESY, Photon Science)**

*KIM, Timur (Diamond Light Source, UK); IWASAWA, Hideaki (Diamond Light Source, UK); DUDIN, Pavel (Diamond Light Source, UK); WATSON, Matthew (University of St. Andrews, UK); LIU, Zhongkai (Shanghai Institute of Technology, China)*

In a metal the Fermi Surface is the separation – in momentum space – between occupied and unoccupied states. Here, thermal excitations are possible and rearrangements of the electronic structures such as the superconducting instability, magnetic moment formation and charge density wave-like phase transitions can be driven by the electronic states on the Fermi surface. Angle-resolved photoelectron spectroscopy (ARPES) can map the Fermi surface of a crystalline solid thanks to conservation of momentum in the photoemission process. The method is highly surface sensitive thus inevitably the momentum component perpendicular to the surface ( $k_{\perp}$ ) is less well resolved than the fully conserved parallel momentum ( $k_{\parallel}$ ). In this presentation we will demonstrate the importance of tuning the total momentum ( $k$ ) and thus  $k_{\perp}$  to precisely determine relevant questions of solid state spectroscopy, such as the precise arrangement of bands in the multi-sheet Fermi surface of FeSe, quasi-one-dimensional  $\text{Ti}_2\text{Mo}_6\text{Se}_6$ , and the distinction between bulk-pockets and surface states in epitaxial films of Gd-doped EuO. As an outlook we will discuss the prospect of performing precise spectral function measurements, a strength of ARPES, across a wide range of photon energies.



## Application of the X-ray Standing Wave Technique to Materials Research

Monday, 15:30  
MW0001  
ID 35

*ALLEGRETTI, Francesco (Physics Department, Technical University of Munich)*

*AUWÄRTER, Wilhelm (Physics Department, Technical University of Munich); BARTH, Johannes V. (Physics Department, Technical University of Munich)*

Normal Incidence X-ray Standing Waves (NIXSW) is a powerful technique based on synchrotron radiation, which can be employed for structural determination of adsorbate-covered surfaces and for investigating the local environment of subsurface diluted species. Due to the specific photon energy range that is commonly needed for these measurements there are only few end-stations available in the world, however the technique has been enjoying a renaissance in the last decade. In this contribution I will describe the fundamentals of the NIXSW technique, its structural sensitivity and special advantages. Moreover, to clarify the range of possible applications I will present some illustrative examples taken from our recent research at the Physics Department (TU Munich) and concerning 1) ultra-thin films of adsorbed metal complexes, 2) monolayers of functional 2D materials, and 3) doping of semiconductor crystals (e.g., titanium dioxide) of photocatalytic interest, respectively.



## Concentration profiles in thin films obtained from Neutron Depth Profiling at the PGAA facility

Monday, 15:45  
MW0001  
ID 4

**TRUNK, Markus**

*GASTEIGER, Hubert A (TUM); Dr GERNHÄUSER, Roman (TUM); GILLES, Ralph; MÄRKISCH, Bastian (Physik Department, TU München); REVAY, Zsolt*

Neutron Depth Profiling (NDP) is a non-destructive, high-resolution, near-surface analytical technique, which measures concentration profiles of a set of light nuclides like He-3, B-10, Li-6, N-14, O-17 [1]. The high neutron capture-flux density of  $3E10 \text{ s}^{-1}\text{cm}^{-2}$  at the PGAA beamline enables good measurement statistics on reasonable time scales and opens the possibility towards tracking changing concentration profiles with a high time resolution [2]. We present the method, show the application in several different materials branches and discuss results from an ex situ study of new electrode coating materials for lithium-ion batteries. Special interest here is the incorporation of passivated lithium in solid-electrolyte-interfaces (SEI), where NDP offers the opportunity to monitor the depth dependent SEI evolution. This project is supported by BMBF 05K16WO1.

[1] R. G. Downing et al., J. Res. Natl. Inst. Stand. Technol. 1993, 98, 109.

[2] Zs. Revay et al., Nucl. Instr. Meth. A 2015, 799, 114-123.



**Monday – September 17<sup>th</sup>, 2018**  
**14:30-16:00**

## ***Micro symposium 1***

# ***In-situ and in-operando studies with special focus on energy materials and catalysis***

**MW1801**

*Each field is linked to the abstract's start page. Just click on the title!*

**14:30-15:00**

*Beatriz Roldan Cuenya*

Operando Insight into Electrocatalytic and Thermal Conversion of CO<sub>2</sub> to valuable Chemicals and Fuels

**15:00-15:15**

*Matthias Bauer*

Development of photon-in/photon-out spectroscopy at PETRA III and applications to studies in catalysis

**15:15-15:30**

*Mirijam Zobel*

In-situ PDF to reveal molecular complexes and track particle formation

**15:30-15:45**

*Dirk Lützenkirchen-Hecht*

Instrumentation for time resolved X-ray absorption spectroscopy at PETRA III

**15:45-16:00**

*Juan J. Velasco-Vélez*

Atmospheric Pressure X-ray Photoelectron Spectroscopy for catalysis and electro-catalysis



## Operando Insight into Electrocatalytic and Thermal Conversion of CO<sub>2</sub> to valuable Chemicals and Fuels

Monday, 14:30

MW1801

ID 440

**ROLDAN CUENYA, Beatriz (Fritz-Haber-Institut der Max-Planck-Gesellschaft)**

Tailoring the chemical reactivity of nanomaterials at the atomic level is one of the most important challenges in catalysis research. In order to achieve this elusive goal, we must first obtain a fundamental understanding of the structural and chemical properties of these complex systems. In addition, the dynamic nature of the nanostructured films and nanoparticle (NP) catalysts and their response to the environment must be taken into consideration. To address the complexity of realworld catalysts, a synergistic approach taking advantage of a variety of cutting-edge experimental methods (EC-AFM, STM, SEM, TPD, AP-XPS, XAFS) has been undertaken. This talk will provide new insights into the thermal hydrogenation and electrocatalytic reduction of CO<sub>2</sub>. Important components missing from most existing studies that we propose to address are the systematic design of catalytically active model NPs (Cu, Cu-Zn, Cu-Ag) with narrow size and shape distributions and tunable oxidation state as well as plasma-activated nanostructured metal films (Ag, Cu), and in situ and operando structural and thermal/electrochemical reactivity characterization under realistic reaction conditions, i.e. at high pressure or under potential control, respectively. The results are expected to open up new routes for the reutilization of CO<sub>2</sub> through its direct conversion into valuable chemicals and fuels such as carbon monoxide, methane, ethylene, methanol, ethanol, and propanol.



### Development of photon-in/photon-out spectroscopy at PETRA III and applications to studies in catalysis

Monday, 15:00  
MW1801  
ID 449

*BAUER, Matthias (Universität Paderborn)*

X-ray absorption spectroscopy (XAS) is nowadays a very established tool to investigate and understand chemical reactions on an atomic level. However, it suffers from particular limitations that hinder for example the identification of metal-reactant interactions in catalytic transformations or the detailed investigation of electronic structures. Nevertheless, hard X-rays are highly advantageous, as they tolerate nearly every experimental environment, as often found in operando studies. Therefore, the development of more sensitive methods, able to resolve the frontier orbital structure of catalysts and with the ability to distinguish coordinating light atoms that are neighbours in the periodic table, are highly needed. High energy resolution fluorescence detected XAS (HERFDXAS), valence-to-core (VtC) and core-to-core (CtC) X-ray emission spectroscopy (XES) offer the opportunity to reach these aims.

In this talk, the presentation of spectrometer types that are funded by the BMBF and implemented at beamline P64 at PETRA III is connected to important results from the field of sustainable chemistry to demonstrate the high potential of such techniques for future applications of socio-economic relevance.



## In-situ PDF to reveal molecular complexes and track particle formation

Monday, 15:15  
MW1801  
ID 439

**ZOBEL, Mirijam (University Bayreuth)**

The pair distribution function (PDF) technique experienced a large revival in recent years in the field of nanomaterials as it is able to access the structure of crystallographically challenging materials. [1] By measuring the total, i.e. the Bragg and diffuse, scattering, a variety of materials comprising liquids, glasses and disordered nanoparticles can structurally be analysed. In-situ/- operando PDF experiments can be carried out with a time resolution of seconds to minutes and the high-energy X-rays (50 – 90 keV) readily penetrate model reactors. This time resolution became possible due to i) the increase in flux at high X-ray energies at synchrotron radiation sources, and ii) novel detector technologies (Pilatus CdTe 1M). Thus recently, not only in-operando insight into batteries under cycling was achieved [2], but we could monitor chemical reactions, bond formation and nucleation processes of nanoparticles. [3] Combined with novel approaches in data processing, we track minute scattering contrasts in-situ to access changes of solvation shells during particle formation.

[1] S. J. L. Billinge, et al., Chem. Commun. 7 (2004) 749

[2] Wiaderek, K. M., et al, J. Am. Chem. Soc. 135 (2013) 4070

[3] M. Zobel, et al., CrystEngComm 18 (2016) 2163



## Instrumentation for time resolved X-ray absorption spectroscopy at PETRA III

Monday, 15:30  
MW1801  
ID 438

**LÜTZENKIRCHEN-HECHT, Dirk (Bergische Universität Wuppertal)**

*BORNMANN, Benjamin (Bergische Universität Wuppertal); Dr MÜLLER, Oliver (SSRL); KLÄS, Jonas (Bergische Universität Wuppertal); FRAHM, Ronald (Bergische Universität Wuppertal)*

After its transformation into a dedicated high brilliance synchrotron source, several efforts have been made to implement the opportunity of time-resolved X-ray absorption spectroscopy (XAS). Thanks to the intense photon beam delivered by a tapered undulator in a wide energy range from about 4-40 keV, a time resolution in the millisecond range appears feasible from theory, needing however dedicated equipment to achieve this challenge. In this presentation, we will summarize the related activities from the past few years. Since the achievable temporal resolution of XAS is limited by the mechanical stability of the monochromator, a design with channel-cut crystals was employed, due to the positive experience with fast-oscillating double-crystal monochromators. The oscillating movement was realized by implementing a direct drive torque motor here, instead of a purely mechanically driven system. The actual Bragg-angle of the monochromator was measured by angular encoders, and in order to cope with the high heat load of the undulator, cryogenic cooling using liquid nitrogen is mandatory. Furthermore, fast detection systems comprising the detector, the read-out electronics as well as spectroscopic amplifiers with a response time in the microsecond range have been developed. Finally, software for data recording and analysis of several thousand EXAFS spectra has been realized. The work presented here is supported by the BMBF, grant numbers 05K13PX1 and 05K10PX1.



## Atmospheric Pressure X-ray Photoelectron Spectroscopy for catalysis and electrocatalysis

Monday, 15:45  
MW1801  
ID 199

**VELASCO-VÉLEZ, Juan J. (FHI der MPG)**

*HÄVECKER, Michael (FHI der MPG); Dr JONES, Travis (FHI der MPG); FREVEL, Lorenz (FHI der MPG); MOM, Rik (FHI der MPG); KNOP-GERICKE, Axel (FHI der MPG); SCHLÖGL, Robert (FHI der MPG)*

The discrepancy between higher operating pressures applied in catalytic processes and lower measurement pressures accessible during surface characterization is known in the community as the “pressure gap”. To bridge this gap, new experimental and instrumental methods have been developed such as Ambient Pressure XPS (AP-XPS), which allows the collection of photoelectrons at pressures in the millibar range. The wealth of information provided by these experimental setups is of prime importance to the understanding of complex systems. However, the study of solid-gas interfaces with partial pressures beyond a few tens of millibars remains a major challenge. Recently, this technique was extended to higher kinetic energy (HAXPES). Notably, this approach combined with a small aperture nozzle increases the operation pressure up to 100 millibars. Here we introduce an atmospheric pressure XP spectroscopy cell based on an array of micro holes coated with graphene that solves the mechanical stability problem of free standing graphene covering large open areas and still provides high transmission for low kinetic energy electrons (higher than 400 eV) combined with a high rate of fabrication reproducibility. We will illustrate the operation of this setup with different examples in liquid and gas phase such as the selective hydrogenation of alkynes into alkenes/alkanes and in the characterization of electrochemical processes on electrified interfaces under aqueous conditions.



**Monday – September 17<sup>th</sup>, 2018**  
**14:30-16:00**

## ***Micro symposium 2***

# ***Structural biology: Instrumentation and methods, current trends and topics***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>14:30-14:55</b> <i>Gerhard Klebe</i> From Fragments to Drug Leads: Combing Crystallography with Docking and Design
<b>14:55-15:11</b> <i>Marialucia Longo</i> In-situ macro-seeding apparatus for protein crystal growth
<b>15:11-15:27</b> <i>Diana Monteiro</i> Rapid mixing microfluidics for time-resolved serial synchrotron crystallography
<b>15:27-15:43</b> <i>Andrea Thorn</i> AUSPEX: a diagnostic tool for graphical X-ray data analysis
<b>15:43-15:58</b> <i>Charlotte Uetrecht</i> Hitting proteins with a sledgehammer-structural characterization with X-rays



## **From Fragments to Drug Leads: Combing Crystallography with Docking and Design**

**Monday, 14:30  
MW0350  
ID 206**

***KLEBE, Gerhard (Inst. pharm. Chemie, Univ. Marburg)***

Fragment-based lead discovery has become increasingly popular to identify first leads for drug development. Advantage of using fragments is their low molecular weight, which leaves sufficient space for subsequent chemical optimization before the pharmaceutically reasonable limit of 500 g/mol is reached. However, the inherent low-binding affinity of fragments poses major challenge to current biophysical and biochemical screening methods and questions whether such techniques can be used as pre-screening filter to reliably detect all putative fragments subsequently to be crystallized with the target protein. Since data collection at synchrotrons has become fast enough, we favor direct fragment screening on protein crystals. We designed a 1270-entry fragment library and tested parts of it against seven different proteins with a mean hit rate of 17%. However, how to optimize weak initial fragment binders to putative drug leads? We developed several design tools to search for analogs, finding larger ligands embedding to original core fragment or allowing a tailored docking of putative side chains to the initial core fragments. Since natural compounds are claimed to be better starting points for drug development, we used the AnalytiCon FRGx fragment collection to discover hits by crystallography. Subsequently, we identified by docking putative lead candidates that embed the core fragment into a larger scaffold available in the 30,000 entry NATx database of natural compounds.



### In-situ macro-seeding apparatus for protein crystal growth

Monday, 14:55  
MW0350  
ID 263

*LONGO, Marialucia (Jülich Centre for Neutron Science JCNS)*

*SCHRADER, Tobias E. (Jülich Centre for Neutron Science JCNS)*

Although X-ray crystallography allowed the determination of the three-dimensional structure of fundamental biomolecules such as proteins and nucleic acids, neutron crystallography provides a powerful complement because hydrogen atoms can be visualized directly. Due to the lower flux of neutrons compared to that of X-rays, bigger crystals (~0.5 mm<sup>3</sup>) are required. So, the improvement of the production of sufficiently large crystals is one of the requisites of neutron crystallography. For this purpose, we aim to design an in-situ macro-seeding apparatus for the growth of sufficiently large protein crystals, where the exchange of the mother liquor is allowed by means of two capillaries attached to a spacer between two flat round glass windows. This spacer defines the volume of the crystallization chamber and will be produced by a 3-D printer. In order to control the crystallization process, a Peltier element will be in thermal contact with the crystallization chamber for temperature control. The designed device allows us to use an inverted microscope to look through the glass windows in order to monitor the crystal size during the crystallization process. The exchange of the mother liquor will allow feeding the growing crystal with fresh, highly concentrated protein in solution. Thereby, the crystal growth does not stop due to a lack of protein in solution.



**Rapid mixing microfluidics for time-resolved serial synchrotron crystallography**

**Monday, 15:11  
MW0350  
ID 289**

***MONTEIRO, Diana (CUI, University of Hamburg)***

*PEARSON, Arwen; Prof. TREBBIN, Martin (University of Hamburg)*

Dynamically resolved, or better known time-resolved, structural studies of proteins rely on specific sample environments at both synchrotrons and XFELs due to two main requirements: the continuous delivery of fresh sample to the X-ray beam and the need to trigger the protein activity. The two most widely used methods for protein triggering are the diffusion of actuators by rapid mixing and light activation using laser pulses. Within collaborative efforts between the Trebbin group and other groups at the University of Hamburg, DESY and ESRF, we have developed sample environments and methods for these complex studies. Specifically, we have recently developed different X-ray compatible devices for serial synchrotron crystallography (SSX) data collection in flow with mixing for which I will present an example using 3D-printed microfluidic devices at beamline ID303-A (ESRF). These devices are capable of delivering ligands to microcrystalline crystal slurries to generate metastable protein species which can then be studied by X-ray diffraction. These versatile methods can be tailored to the requirements of the protein target characteristics such as time scales of reaction and sample. Therefore, the development of general and user-friendly sample environments is an important step in making these experiments more widely available.



## AUSPEX: a diagnostic tool for graphical X-ray data analysis

Monday, 15:27  
MW0350  
ID 394

*THORN, Andrea (Julius-Maximilians-Universität Würzburg)*

*PARKHURST, James (Diamond Light Source, Didcot, UK); EMSLEY, Paul (MRC Laboratory of Molecular Biology, Cambridge, UK); NICHOLLS, Robert (MRC Laboratory of Molecular Biology, Cambridge, UK); VOLLMAR, Melanie (Diamond Light Source, Didcot, UK); EVANS, Gwyndaf (Diamond Light Source, Didcot, UK); MURSHUDOV, Garib (MRC Laboratory of Molecular Biology, Cambridge, UK)*

Ice diffraction is a much bigger and more widespread problem in protein crystallography than commonly recognized [1]. 'Ice rings' are notoriously difficult to account for in integration [2]. The resulting experimental structure factor errors can affect the modelling of macromolecular structures and may even prevent their solution. Furthermore, ice rings are hard to recognize from diffraction images produced by modern pixel detectors, and even harder to identify in already processed data [1]. In order to address this problem, a new software tool, AUSPEX, has been developed. Here, it will be demonstrated how AUSPEX facilitates the detection of common problems in data collection and processing [3], allowing for visual inspection of the observed intensity (or amplitudes) distribution versus resolution and automatic ice ring detection. AUSPEX can also be used to investigate the structure and distribution of errors within crystallographic data sets; this reveals a number of additional pathologies in the processing and conversion of X-ray and neutron data. AUSPEX will be available via CCP4 and as a web service.

[1] Thorn, A., Parkhurst, J.M., Emsley, P., Nicholls, R., Vollmar, M., Evans, G. & Murshudov, G.N. (2017) Acta Cryst D73, 729-737.

[2] Parkhurst, J.M., Thorn, A., Vollmar, M., Winter, G., Waterman, D.G., Fuentes-Montero, L.,

Gildea, R.J., Murshudov G.N. & Evans, G. (2017), IUCrJ 4, 626-638.

[3] Garman, E. F. & Mitchell, P. E. (1994) J. Appl. Cryst. 27, 1070-1074.



## **Hitting proteins with a sledgehammer - structural characterization with X-rays**

**Monday, 15:43  
MW0350  
ID 428**

***UETRECHT, Charlotte (Heinrich Pette Institute & European XFEL)***

Native ion mobility mass spectrometry (MS) is a perfect tool to study protein complexes in a mass and conformation specific manner. Despite remarkable sensitivity and selectivity the structural resolution is limited in native MS. On the other hand, it allows monitoring structural transitions, which proteins and protein complexes undergo, e.g. during the viral life-cycle. However, such transient states cannot be purified and are inaccessible for crystallography. The European XFEL, the world's most intense hard X-ray free-electron laser (XFEL), has just become operational and offers an opportunity to obtain high resolution structures of single particles. The benefits of native MS for single particle imaging of transient intermediates at European XFEL and initial feasibility studies on achievable ion flux will be presented. Furthermore, we have tested native MS in conjunction with the soft XFEL FLASH and PETRA P04 soft X-ray synchrotron radiation. Our recent results show potential for soft X-rays protein complex fragmentation and dissociation for structural proteomics. The available intensity could alleviate proposed upper size limits for UV photo dissociation of protein complexes.

**Monday – September 17<sup>th</sup>, 2018**  
**16:30-18:00**

## **Poster session 1**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>ID</b>	<b>Title</b>	<b>Presenter</b>
3	Design study of the new HiCoReLAN detector	<i>Gregor Nowak</i>
7	In situ studies at elevated temperatures on CoRe alloys for ultra-high temperature applications using X-ray/neutron diffraction and small-angle neutron scattering	<i>Ralph Gilles</i>
15	The Rhizosphere Physical Network	<i>Pascal Benard</i>
33	Morphological evolution of gold nanostructures on an inorganic semiconductor quantum dot array with time-resolved GISAXS	<i>Neelima Paul</i>
34	An X-ray and neutron scattering study of self-organized anodic TiO <sub>2</sub> nanotubes for battery applications	<i>Neelima Paul</i>
36	Dynamics and structure of PNIPAM-based microgels with different internal homogeneity	<i>Tetyana Kyrey</i>
37	The order/disorder transformation of $\beta$ phase in binary and ternary $\gamma$ TiAl based alloys studied by synchrotron and neutron diffraction	<i>Victoria Kononikhina</i>
39	Breakdown of helical order in the Dzyaloshinskii-Moriya Antiferromagnet Ba <sub>2</sub> CuGe <sub>2</sub> O <sub>7</sub>	<i>Sebastian Muehlbauer</i>
41	Shack-Hartmann sensors for X-ray multicontrast imaging	<i>Anton Plech</i>
45	Neutron Depth Profiling at a focused neutron beam to study Li-ion transport in thin-film batteries	<i>Egor Vezhlev</i>
48	Bringing Neutrons to the User – The HBS Project for Accelerator Based Neutron Sources	<i>Thomas Gutberlet</i>
49	Soft X-ray analysis of microgels and polymer-based microcontainers in aqueous environment	<i>Rainer Fink</i>
50	Incommensurate magnetic systems studied with the three-axis spectrometer MIRA	<i>Robert Georgii</i>
51	CSPEC - the cold time-of-flight spectrometer for the ESS	<i>Wiebke Lohstroh</i>
52	N-doping of Niobium: In-situ EXAFS experiments	<i>Jonas Kläs</i>
53	Magnetic protein separation with new affinity tags for bare iron oxide nanoparticles	<i>Sebastian Schwaminger</i>
56	Water swelling and exchange kinetics in multi-stimuli responsive PNIPAM-based block copolymer thin films	<i>Lucas Kreuzer</i>



57	Structure Determination of a new Molecular White-Light Source	<i>Benjamin Klee</i>
59	Growth and characterization of $\text{Fe}_3\text{O}_4/\text{Nb}:\text{SrTiO}_3$ heterostructure	<i>Anirban Sarkar</i>
61	Magnetization and Magnetocaloric Effect in $\text{MnFe}_4\text{Si}_3$	<i>Nour Maraytta</i>
62	Simulation of grazing-incidence small-angle scattering for soft matter studies	<i>Marina Ganeva</i>
70	POLI: Multifunctional Single Crystal Diffractometer with Polarized Neutrons at MLZ	<i>Hao Deng</i>
71	Time resolved diffraction of SHS reactions at the HED instrument at European XFEL	<i>Wolfgang Morgenroth</i>
73	Neutron reflectivity investigation on thermo-responsive copolymers with embedded magnetic nanoparticles	<i>Senlin Xia</i>
80	Ferrimagnetic films investigated by polarized neutron channeling in planar waveguides	<i>Florin Radu</i>
88	Morphology–Function Relationship of Thermoelectric Nanocomposite Films from PEDOT:PSS with Silicon Nanoparticles	<i>Nitin Saxena</i>
92	Defect Imaging Using the Positron-Microbeam of the CDB Spectrometer at NEPOMUC	<i>Thomas Gigl</i>
93	Development of a Sample Environment for in-situ Dynamic Light Scattering in Combination with Small Angle Neutron Scattering for the Investigation of Soft Matter at the European Spallation Source ESS	<i>Andreas Josef Schmid</i>
94	Temperature Induced Structural Evolution of DMPC-Saponin-Mixtures: From Bicellar to Vesicular Structures?	<i>Carina Dargel</i>
95	Nanoscience crystallography at a high brilliance laboratory X-ray diffractometer: from mesoscopic to interatomic length scales	<i>Emmanuel Kentzinger</i>
96	Theoretical spin-wave dispersions in the antiferromagnetic phase AF1 of $\text{MnWO}_4$ based on the polar atomistic model in P2	<i>Benqiong Liu</i>
98	Spatially resolved composition and functionality of thin film solar cells	<i>Maurizio Ritzer</i>
100	Printed films of conjugated high-efficiency polymers and small acceptor molecules	<i>Kerstin Wienhold</i>
102	Proton exchange membranes for fuel cell applications studied with the Pulsed Low Energy Positron System (PLEPS)	<i>Mahmoud Gomaa</i>
103	Magnetic structures and proton dynamics in phosphatic oxyhydroxides	<i>SoHyun Park</i>
104	Restructuring of organic solvents around iron oxide nanoparticles capped by oleic acid	<i>Sabrina Thomä</i>
107	Metal to Nonmetal Phase Separation in Expanded liquid Rubidium	<i>Wolf-Christian Pilgrim</i>
108	The new Chemical Crystallography Beamline P24 at Petra III (PEX-E), Desy	<i>Carsten Paulmann</i>
110	Rotatable load frames for neutron diffraction – analysis of strain, texture, phase transformations and elastic constants	<i>Markus Hoelzel</i>
113	The new Neutron Depth Profiling instrument at the Prompt Gamma Activation Analysis facility of MLZ	<i>Lukas Werner</i>
115	Energy research with neutrons (ERWIN) and installation of a neutron powder diffraction option at MLZ	<i>Michael Heere</i>
118	BATS – a highly flexible option for inverted TOF spectroscopy on the neutron backscattering spectrometer IN16B	<i>Markus Appel</i>
120	Quantitative investigation of the “surface trans-effect” in an adsorbed metal-organic complex by means of X-ray Standing Waves	<i>Peter S. Deimel</i>
121	Nuclear resonance scattering polarimetry on single crystals of iron spin crossover compounds	<i>Lena Scherthan</i>
122	Development of new testing machine (load and temperature) for the investigation of superalloys	<i>Johannes Munke</i>



123	Structural investigations on PEDOT:PSS polymeric electrodes by means of GISAXS and GIWAXS	<i>Lorenz Bießmann</i>
126	The MEPHISTO beam port for nuclear and particle physics	<i>Jens Klenke</i>
127	Pore filling mechanism in the chemically stable Metal-Organic Framework DUT-67(Zr) by Neutron Powder Diffraction	<i>Volodymyr Bon</i>
129	The beneficial effect of Rubidium in Cu(In,Ga)Se <sub>2</sub> solar cells	<i>Claudia S. Schnohr</i>
131	Nanometer and sub-nanometer layer reference samples for X-ray fluorescence - New results and applications	<i>Markus Krämer</i>
133	Near-surface region characterization of nitrogen treated single-crystal Nb (100)	<i>Guilherme Dalla Lana Semione</i>
134	Neutron Optics from NOB Nano Optics Berlin	<i>Thomas Krist</i>
136	P65 – a workhorse beamline for in-situ and operando XAFS spectroscopy at the PETRA III storage ring	<i>Edmund Welter</i>
137	FIREPOD – the fine resolution powder diffractometer @ Berlin research reactor BER II	<i>Alexandra Franz</i>
139	High-Flux XAFS-Beamline P64 at PETRA III	<i>Wolfgang Caliebe</i>
143	Beamline P02.1: A Workhorse for High-Resolution Powder Diffraction & Total Scattering Experiments at PETRA III, DESY	<i>Michael Wharmby</i>
149	Heterogeneous local order in self-assembled nanoparticle films revealed by X-ray cross-correlations	<i>Felix Lehmkuhler</i>
150	Zone-plate based soft x-ray microscopy with sub-10 nm resolution	<i>Rainer Fink</i>
154	Critical scattering in classical and nearly quantum critical systems	<i>Heiko Trepka</i>
155	Modeling of small-angle scattering data from proteins with modifications	<i>Martin Schmiele</i>
157	Operando surface X-ray diffraction studies of electrodeposited Co oxide thin films during oxygen evolution	<i>Jochim Stettner</i>
162	New setup for in-situ MBE growth studies using high energy x-ray diffraction	<i>Joachim Wollschläger</i>
163	In-situ Microspectrophotometry for Macromolecular Crystallography	<i>Thomas Hauß</i>
164	Upgrade of the NEPOMUC remoderator	<i>Marcel Dickmann</i>
165	New possibilities for Hard X-Ray Photoelectron Spectroscopy at DESY	<i>Christoph Schlueter</i>
166	Sample environment and Applications of the Hot Neutron Single Crystal Diffractometer HEiDi@MLZ	<i>Martin Meven</i>
168	The interaction of viral fusion peptides with model lipid membranes at high hydrostatic pressure	<i>Julia Nase</i>
169	The new high-resolution neutron spin-echo spectrometer at MLZ: J-NSE Phoenix	<i>Stefano Pasini</i>
170	High-temperature stability of matrix in boron-containing Co-Re-Cr alloys for gas turbine applications	<i>Pavel Strunz</i>
172	Investigating the interaction of water with vanadium oxide surfaces by XPS	<i>Dana Goodacre</i>
173	Low-temperature phase transitions in PrAlO <sub>3</sub> -SrTiO <sub>3</sub> series	<i>Leonid Vasylechko</i>
175	Laser-neutron pump-probe experiment at the neutron time-of-flight spectrometer TOFTOF	<i>Dominik Schwaiger</i>
176	Nanostructured TiO <sub>2</sub> /SnO <sub>2</sub> Templated by Amphiphilic Block Copolymer for Lithium-Ion Battery Anodes	<i>Shanshan Yin</i>
179	Influence of benzocaine and propranolol on phospholipid bilayers	<i>Gaetano Mangiapia</i>
180	Formation and growth of mesoglobules in aqueous poly (N-isopropylacrylamide) solutions revealed with fast pressure jumps	<i>Bart-Jan Niebuur</i>
185	Crystal structure dependent solidification of undercooled glass-forming melts	<i>Fan Yang</i>



188	Nuclear inelastic scattering for identification of iron ligand modes in dinitrosyl iron complexes and nitrogenase single crystals	<i>Christina S. Müller</i>
192	Observing the structural evolution during chemical processes in-situ via SANS	<i>Dominic Hayward</i>
194	A split-and-delay unit for the European XFEL: Enabling hard x-ray pump/probe experiments at the HED instrument	<i>Sebastian Roling</i>
195	Scanning X-ray Diffraction Microscopy of Ion Irradiated VO <sub>2</sub>	<i>Andreas Johannes</i>
197	Operando X-ray absorption spectroscopy (XAS) study of the selective CO methanation on Ru/TiO <sub>2</sub> catalysts: TiO <sub>2</sub> surface morphology effects	<i>Shilong Chen</i>
198	Morphology of printed active layers for organic solar cells as studied with advanced scattering techniques	<i>Xinyu Jiang</i>
202	The low flux neutron source AKR-2	<i>Nico Bernt</i>
203	Characterization of organic thin films for potential application in energy conversion devices	<i>Christian Weindl</i>
205	Supramolecular structure of monohydroxy alcohols	<i>Jennifer Bolle</i>
209	SAPHiR, the instrument for neutron science at high P and T	<i>Nicolas Walte</i>
213	GISAXS reconstruction of grating profiles produced using self-aligned multiple patterning	<i>Mika Pflüger</i>
215	Effect of inorganic SnIP nanoparticles on the morphology of polymer blends for photovoltaic applications	<i>Sebastian Grott</i>
220	Spin dynamics and anomalous anisotropy gap in the metallic perovskite SrRuO <sub>3</sub>	<i>Kevin Jenni</i>
225	Morphology Control of Low Temperature Fabricated ZnO Nanostructures for Transparent Active Layers in All Solid-State Dye-Sensitized Solar Cells	<i>Kun Wang</i>
226	Precision determination of the axial-vector coupling constant from neutron beta decay	<i>Bastian Märkisch</i>
227	Role of native oxide layer in silicon anodes for Li/S batteries	<i>Luca Silvi</i>
229	Colloidal quantum dots solids for photovoltaics	<i>Wei Chen</i>
234	Specialized Sample Environment at BESSY II	<i>Dirk Wallacher</i>
236	X-ray magnetic linear dichroism as a probe for non-collinear magnetic states in a DyCo <sub>5</sub> single ferrimagnetic layer	<i>Florin Radu</i>
245	Combining x-ray emission and x-ray Raman spectroscopy for the study of Earth materials at high pressure and high temperature	<i>Christian Sternemann</i>
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248	Complementarity of Neutrons and X-rays on the Example of Hydrocarbon/Fluorocarbon Small Unilamellar Vesicles	<i>Anja Franziska Hörmann</i>
251	Resonant X-ray Diffraction at the Petra III Chemical Crystallography Beamline	<i>Melanie Nentwich</i>
254	Investigation of organically linked iron oxide nanoparticle supercrystals using SAXS and SANS	<i>Malte Blankenburg</i>
255	Temperature dependent study of the local structure of bromine ions in polymerized ionic liquids	<i>Mirko Elbers</i>
258	Inelastic neutron scattering studies of magnons in the conical and field-polarized phase of MnSi	<i>Lukas Beddrich</i>
259	Ultra small iron nanoparticle superlattice on graphene on iridium	<i>Konstantin Krausert</i>
260	Hard x-ray photoemission spectroscopy of in operando strained Vanadiumdioxide films on PMN-PT	<i>Arndt Quer</i>
261	Exchange coupling effects in hybrid Gr-4f RE systems	<i>Leticia De Melo Costa</i>
262	Exhaustive X-ray Crystallographic Screening of a Hit-Enriched 96 Fragment Library Against Diverse Targets	<i>Francesca Magari</i>



266	Investigation of metastable precipitates in Ti-15Mo by in-situ SANS	Vasyl Ryukhtin
268	Backfilling of Mesoporous Titania Structures with Heavy Element Containing Small Molecules and High-Efficiency Polymer PTB7-th	Raphael Märkl
273	Formation of a micrometer positron beam at the Scanning Positron Microscope	Johannes Mitteneder
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290	Residual Stress Characterization of Friction Surfacing Coated AA2024 Plates	Gleb Dovzhenko
291	Structural Investigations of a Model System for Multiple Sclerosis	Benjamin Krugmann
294	Moisture-Induced Degradation in Perovskite-Based Photovoltaics	Lennart Reb
295	Sharper X-ray vision through aberration-corrected optics	Frank Seiboth
296	Internal stress in electrical steel sheets – Effects and Applications	Tobias Neuwirth
298	Implanter applications of polyatomic ions from a high current liquid metal alloy ion source	Lothar Bischoff
299	Exploring dynamic structures of bionanocages by SRCD	João Jacinto
302	Fast Detectors and Electronics for Nuclear Resonant Scattering of Synchrotron Radiation	Olaf Leupold
306	Transverse Free-Electron Target for the Heavy-Ion Storage Ring CRYRING@ESR	Stefan Schippers
307	GISAXS investigation of highly ordered nanostructures	Wolfgang Gruber
309	Internal dynamics of different folding intermediates of apo-myoglobin	Livia Balacescu
310	Reference-free GIXRF-XRR based characterization of nanolayers and nanostructures	Yves Kayser
311	Efficient X-ray optics for spectroscopy of highly charged ions	Robert Löttsch
312	Neutron Depth Profiling with Highest Luminosity Pulsed Beams	Roman Gernhäuser
313	P66 beamline for time-resolved luminescence experiments under VUV excitation at PETRA synchrotron	Aleksei Kotlov
317	Study of Magnetic Dumbbell nanoparticles using advanced scattering techniques	Nileena Nandakumaran
318	Generation of high-density positron pulses at NEPOMUC for production of an electron-positron plasma	E. V. Stenson
328	3D Material Characterization by Laminographic Imaging: Status and Prospects	Tilo Baumbach
330	Upgrades at SPHERES	Michaela Zamponi
334	Phase Retrieval for X-Ray Near-field Holography beyond Linearisation	Johannes Hagemann
335	Neutron structure analysis of NADH cytochrome b5 reductase	Yu Hirano
340	Cryptoferromagnetic-like effect in Gd/Nb superlattice	Yury Khaydukov
342	SI traceable characterization of nanomaterials by X-ray spectrometry	Burkhard Beckhoff
344	DMPC model membrane structure and dynamics in the presence of the saponin aescin	Thomas Hellweg
351	In situ triaxial deformation experiments on a sandstone sample for strain investigation at the neutron time-of-flight diffractometer EPSILON	Christian Scheffzük
359	Structural investigation of $\text{Li}_6\text{PS}_5\text{Cl}$ for applications in all-solid-state batteries	Ruth Giesecke
360	SKADI - Small-Angle Neutrons Scattering at the ESS	Sebastian Jaksch
362	In-situ neutron scattering study of adsorption induced structural deformation in CAU metal organic frameworks – can structural contraction increase hydrogen uptake?	Margarita Russina



365	Thermally induced cation re-ordering in activated Li- and Mn-rich layered oxide Li-ion cathode materials	<i>Florian Sigel</i>
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390	Temperature Sample Environment for the High Field Magnet at the Helmholtz-Zentrum Berlin	<i>Klaus Kiefer</i>
391	Interfacial spin-coupling in magnetic bilayers and their role for ultrafast magnetization dynamics	<i>Markus Gleich</i>
392	Vacuum-compatible hybrid photon counting pixel detector for WAXS, XRD and XRR in the tender X-ray range	<i>Dieter Skroblin</i>
400	Study of the crystalline structure and oxidation states of the Co and Pr of the serie $\text{La}_{0.5-x}\text{Pr}_x\text{Ba}_{0.5}\text{CoO}_{3-\delta}$ with synchrotron radiation	<i>Diana Garcés</i>
402	Photothermal radiometry study of heavy ion beam induced modification of thermal properties of graphite	<i>Christina Trautmann, Marilena Tomut</i>
404	RESEDA: Resonance Spin Echo Spectrometer	<i>Olaf Soltwedel</i>
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418	High-Pressure Hydrogen Cell for in-situ Characterisation of Hydrogen Storage Materials	<i>Neslihan Aslan</i>
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423	Fragment Screening on Protein Kinase A and PIM1-Kinase	<i>Andreas Heine</i>
436	Pulsed Laser Deposition setup for in-situ experiments with at neutron and synchrotron sources.	<i>Alexander Goikhman</i>
442	Structure and Dynamics of Confined Liquids Studied by an X-ray Surface Force Apparatus	<i>Markus Mezger</i>

## Design study of the new HiCoReLAN detector

ID 3

**NOWAK, Gregor (Helmholtz-Zentrum Geesthacht)**

*BURMESTER, Jörg (Helmholtz-Zentrum Geesthacht); PLEWKA, Jörn (Helmholtz-Zentrum Geesthacht); CHRISTIAN, Jacobsen (Helmholtz-Zentrum Geesthacht); BELDOWSKI, Andreas (Helmholtz-Zentrum Geesthacht); GREGERSEN, Carsten (Helmholtz-Zentrum Geesthacht); HEDDE, John (Helmholtz-Zentrum Geesthacht); FENSKE, Jochen (Helmholtz-Zentrum Geesthacht); MÜLLER, Martin (Helmholtz-Zentrum Geesthacht)*

Modern neutron Multi-Wire-Proportional-Chambers operating with alternative solid-state converter as  $^{10}\text{B}_4\text{C}$  coatings have the potential to surpass the position resolution and count rate capability of  $^3\text{He}$  based detectors at comparable detection efficiency [1, 2]. The use of large area converter coatings on sub-mm substrates makes it essential to develop a mechanical concept to avoid their deformations in operation due to their own weight and acting electrostatic forces resulting from the applied HV for gas amplification. HZG has introduced [1] and investigated the idea of stabilizing the converter elements by gas pressure gradient between both sides of the converter to counteract these forces. A gas vessel-free PSD consisting of 24× parallel stacked converters with a detection depth precision <12 mm was designed for a position resolution of 2 mm. The deposition method of  $^{10}\text{B}_4\text{C}$  coatings with thicknesses up to 10  $\mu\text{m}$  on pretreated Al substrates was elaborated [2, 3]. The delay-line read-out of the detector is designed for count rates up to >200kc/s per detector plane. First neutron tests at the ESS test beamline V20 of the new read-out chain connected to a small  $^{10}\text{B}_4\text{C}$  neutron prototype detector verified the envisaged signal to noise ratio.

[1] European Patent: EP 17184906.0 (filed at 04.08.2017)

[2] European Patent Application 2 997 174 (14.07.2014)

[3] G. Nowak et al., J. Appl. Phys. 117, 034901 (2015)

## In situ studies at elevated temperatures on CoRe alloys for ultra-high temperature applications using X-ray/neutron diffraction and small-angle neutron scattering

ID 7

**GILLES, Ralph**

*MUKHERJI, Debashis (TU Braunschweig); KARGE, Lukas; STRUNZ, Pavel (Nuclear Physics Institute); BERAN, Premysl (Physical Nuclear Institute); KRIELE, Armin (Helmholtz Zentrum Geesthacht); HOFMANN, Michael; Prof. RÖSLER, Joachim (TU Braunschweig)*

Ni-base superalloys, well known as high-temperature alloys, are used in gas turbines (both stationary for electric power generation and in aircraft). They fulfill the requirements of high-temperature strength, ductility, corrosion resistance and high creep resistance needed in these applications and have a stable microstructure. Due to the fact, that the service temperature already reached 80% of the Ni melting point, new alloy systems in industry are under consideration to substitute Ni-base superalloys in future. In this contribution CoRe based alloys will be presented as a potential candidate for future applications [1]. Why CoRe alloys? Co-based alloys are already in use as gas turbine material in static components like turbine vanes or in combustor sections because of their intrinsic properties and the ease of manufacturing. The melting point of Co alloys can be considerably enhanced with Re additions (Re has the third highest melting point in the periodic table). The binary Co and Re system is isomorphous with an hcp structure at room temperature. Additions of Ta, C, Cr and B lead to further improvements of the properties in the CoRe alloy. The main contributor to the strength of the CoRe alloy are fine TaC precipitates with sizes below 100 nm which are homogeneously distributed in the alloy. In-situ experiments at very high temperatures (up to 1500°C) with neutron scattering techniques have been used in this study because of the high penetration ability of neutrons and the large neutron beam cross section (about 1 cm<sup>2</sup>). Consequently, these kinds of measurements obtain a large volume representative bulk result from the investigated CoRe alloys [2].

[1] J. Rösler, D. Mukherji, T. Baranski (2007), Adv. Eng. Mater 9, 876-881.

[2] R. Gilles, D. Mukherji, L. Karge, P. Strunz, P. Beran, B. Barbier, A. Kriele, M. Hofmann, H. Eckerlebe, J. Rösler, J. Appl. Cryst. (2016), 49, 1253-1265.



## The Rhizosphere Physical Network

ID 15

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Plant roots release a highly polymeric blend of substances called mucilage into the soil. Various alterations of soil hydraulic properties in their vicinity (the rhizosphere) have been attributed to the presence of mucilage. Despite its definition as a hot spot of microbial activity and the immense amounts of water (about 40% of all terrestrial precipitation) that cross the rhizosphere the mechanisms how mucilage affects soil hydraulic properties remains unclear. Mucilage adsorbs water, reduces its surface tension and increases its viscosity. A consequence is the formation of characteristic structures like long filaments (at low concentration) and twodimensional structures (at high concentration) that span throughout the porous medium during soil drying. The spatial configuration of water impacts the retention and transport properties of the rhizosphere. During drying, mucilage polymers become increasingly viscous and do not move as fast as the receding water phase, thereby acting as a network holding water and increasing the water retention capacity. This viscous network also increases the connectivity of the liquid phase, which might maintain water and solute transport in drying soils – i.e. the relative unsaturated conductivity drops more gradually. In this study the spatial distribution of dry mucilage structures was resolved via synchrotron micro X-ray CT. Results provided the basis for our concept of how mucilage affects the soil hydraulic properties of the rhizosphere.

## Morphological evolution of gold nanostructures on an inorganic semiconductor quantum dot array with time-resolved GISAXS

ID 33

**PAUL, Neelima**

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In optoelectronic devices based on quantum dot arrays, thin nanolayers of gold are preferred as stable metal contacts and for connecting recombination centers. The optimal morphology requirements are uniform arrays with precisely controlled positions and sizes over a large area with long range ordering since this strongly affects device performance. To understand the development of gold layer nanomorphology, the detailed mechanism of structure formation are probed with timeresolved grazing incidence small-angle X-ray scattering (GISAXS) during gold sputter deposition. Gold is sputtered on a CdSe quantum dot array with a characteristic quantum dot spacing of  $\approx 7$  nm. In the initial stages of gold nanostructural growth, a preferential deposition of gold on top of quantum dots occurs. Thus, the quantum dots act as nucleation sites for gold growth. In later stages, the gold nanoparticles surrounding the quantum dots undergo a coarsening to form a complete layer comprised of gold-dot clusters. Next, growth proceeds dominantly via vertical growth of gold on these gold-dot clusters to form a gold capping layer. In this capping layer, a shift of the cluster boundaries due to ripening is found. Thus, a templating of gold on a CdSe quantum dot array is feasible at low gold coverage.

N. Paul, E. Metwalli, Y. Yao, M. Schwartzkopf, S. Yu, S. V. Roth, P. Müller-Buschbaum, A. Paul, *Nanoscale* 7 (2015) 9703-9714.

## An X-ray and neutron scattering study of self-organized anodic TiO<sub>2</sub> nanotubes for battery applications

ID 34

**PAUL, Neelima**

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Self-organized anodic titania (TiO<sub>2</sub>) nanotube arrays are an interesting model anode material for use in Li-ion batteries owing to their excellent rate capability, their cycling stability and their enhanced safety compared to graphite. A composite material where carbothermally treated conductive TiO<sub>2</sub> nanotubes are used as support for a thin silicon film has been shown to have the additional advantage of high lithium storage capacity. This article presents a detailed comparison of the structure, surface and bulk morphology of self-organized conductive TiO<sub>2</sub> nanotube arrays, with and without silicon coating, using a combination of X-ray diffraction, X-ray reflectivity, grazing incidence small angle X-ray scattering (GISAXS) and time-of-flight grazing-incidence small angle neutron scattering (TOF-GISANS) techniques. X-ray diffraction shows that the nanotubes crystallize in the anatase structure with a preferred (004) orientation. GISAXS and TOF-GISANS are used to study the morphology of the nanotube arrays, delivering values for the inner nanotube radius and intertubular distances with high statistical relevance because of the large probed volume. The analyses reveal the distinct signatures of a prominent lateral correlation of the TiO<sub>2</sub> nanotubes of ~94 nm and a nanotube radius of ~46 nm. The porosity averaged over the entire film using TOFGISANS is 46%. The inner nanotube radius is reduced to half (~23 nm) through the silicon coating, but the prominent lateral structure is preserved. Such in-depth morphological investigations over large sample volumes are useful for the development of more efficient battery electrode morphologies.

Paul N., Brumbarov J., Paul A., Chen Y., Moulin J.-F., Müller-Buschbaum P., Kunze-Liebhäuser J., Gilles R., Journal of Applied Crystallography, 48 (2015) 444 – 454.

## Dynamics and structure of PNIPAM-based microgels with different internal homogeneity

ID 36

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*WITTE, Judith (TU Berlin); VON KLITZING, Regine (TU Darmstadt); WELLERT, Stefan (TU Berlin); HOLDERER, Olaf*

Due to the reversible temperature induced volume phase transition (VPT), PNIPAM microgels serve as model systems for basic research. Moreover, the variation of the chemical composition tunes the functionality and responsivity. Changing the monomer feeding process influences homogeneity of the polymer particles and changes structure and dynamics of such systems. The classical particles preparation by means of precipitation polymerization leads to a core-shell structure of polymer particles, whereas continuous monomer feeding process leads to a more homogeneous structure. In this contribution we present neutron spin-echo (NSE) and small-angle neutron scattering (SANS) experiments of inhomogeneously and homogeneously cross-linked networks of same chemical composition of PNIPAM-based microgels. Frozen inhomogeneities and density fluctuations have been observed in addition to Zimm like segmental polymer dynamics depending on the homogeneity of the particle and the cross-link density. Details of the internal microgel structure and its dependence on different synthesis parameters have been obtained in this way.

## The order/disorder transformation of $\beta$ phase in binary and ternary $\gamma$ TiAl based alloys studied by synchrotron and neutron diffraction

ID 37

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Due to their high melting point, low density, and good oxidation resistance,  $\gamma$ -TiAl based alloys have recently started to replace Ni-based superalloys as a material for turbine blades in aircraft engines [1]. Conventional TiAl alloys usually contain the ordered phases  $\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al at lower temperatures and disordered  $\alpha$ -Ti(Al) phase at higher temperatures. Additional alloying elements like Nb, Mo, Ta, Cr or Fe, can stabilize the disordered  $\beta$ -Ti(Al) phase (A2 structure), which can transform at lower temperatures to ordered  $\beta_0$ -TiAl (B2 structure) or even to more complex phases like  $\tau_2$  or  $\omega$ . The ductile body centered cubic (bcc)  $\beta$  phase is important for processing because it significantly improves the hot forming behaviour of the material. Otherwise the ordered low temperature  $\beta_0$  phase is said to embrittle the material at service temperature. Unfortunately little is known about the exact order/disorder transformation temperatures of  $\beta/\beta_0$  in several ternary alloy systems and the influence of  $\beta$  stabilizing elements is still under research. Additionally, even for the binary TiAl phase diagram the existence of an ordered  $\beta_0$  phase field at high temperatures has yet not been finally proofed or rebutted [2]. With conventional in situ investigation methods like differential scanning calorimetry (DSC) an unambiguous assignment of a certain peak to the  $\beta_0 \leftrightarrow \beta$  transformation is not possible in TiAl alloys. X-ray diffraction (XRD) measurements are also not suitable because the superstructure reflections are very weak due to the small electron density differences of the different atom sites in the ordered crystal structures. However in situ neutron diffraction (ND) is most suited to study order/disorder transformations in titanium aluminides [3,4]. The neutron scattering lengths of Ti and Al are almost equal in magnitude but of opposite sign. Thus disordered phases, with a Ti:Al ratio close to one, yield only very weak diffraction peaks, because the average scattering length is almost zero. The fundamental reflections in ordered TiAl crystal structures are also very weak because the scattering lengths of Al sites and Ti sites with their opposite sign add up. However the superstructure reflections of these ordered TiAl crystal structures become rather large, because they contain the difference of the scattering lengths of each site.

*(shortened by editors)*



## Breakdown of helical order in the Dzyaloshinskii-Moriya Antiferromagnet Ba<sub>2</sub>CuGe<sub>2</sub>O<sub>7</sub>

ID 39

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Neutron diffraction on a triple-axis spectrometer and a small-angle neutron scattering instrument is used to study the magnetic phase transition in tetragonal Ba<sub>2</sub>CuGe<sub>2</sub>O<sub>7</sub> at zero magnetic field. In addition to the incommensurate cycloidal antiferromagnetic (AFM) long-range order, we establish that weak incommensurate ferromagnetism (FM) also arises below the transition temperature  $T_N$  identified by sharp Bragg peaks close to the  $\Gamma$  point. The intensities of both the incommensurate AFM and FM Bragg peaks vanish abruptly at  $T_N$ , which is indicative of a weak first-order transition. Above  $T_N$ , evidence is presented that the magnetic intensity within the tetragonal (a; b) plane is distributed on a ring in momentum space whose radius is determined by the incommensurate wave vector of the cycloidal order. We speculate that the associated soft fluctuations are at the origin of the weak first-order transition in the spirit of a scenario proposed by Brazovski. [1]

[1]Phys. Rev. B 96, 134409 (2017)

## Shack-Hartmann sensors for X-ray multicontrast imaging

ID 41

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Beam-modulation techniques in X-ray imaging can serve for disentangling different contrast modalities, such as absorption, phase, and dark-field contrast. These encompass Talbot-grating interferometry of shadow mask (Hartmann) techniques. We have devised micro-lens arrays to be used as Shack Hartmann masks for dose-efficient, single-exposure imaging of dynamic processes. The Shack-Hartmann mask consists of a 2D array of microlenses that partition a wide X-ray beam into individual beamlets, whose attenuation, deviation and broadening will reflect sample properties like absorption, phase shift and scattering power, respectively. Thus, an efficient use of the incoming X-ray flux is made, which allows reducing exposure times down to few micro-seconds. This approach was used to image the structure formation process during pulsed laser ablation in liquids (PLAL). PLAL is a procedure to produce nanoparticles suspended in liquids by ablating a target with an intense pulsed laser beam. The product relies on an intricate interplay between different length and time scales. We will show that different lens arrays as a 3D-printed crossed cylinder lens array (SHARX [1]) or a compound lens array (CARL [2]) of structured polyimide foils are capable of delivering information on macroscopic dynamics as well as the occurrence of nanoparticulate mass during the process.

1. T. dos Santos Rolo, S. Reich, D. Karpov, S. Gasilov, D. Kunka, T. Baumbach, A. Plech: A Shack-Hartmann sensor for single-shot multi-contrast imaging with hard X-rays, arXiv: 1802.10045 (2018).
2. S. Reich, T. dos Santos Rolo, A. Letzel, T. Baumbach, A. Plech: Scalable, large area compound array refractive lens for hard X-rays, Appl. Phys. Lett. 112, 151903 (2018)

## Neutron Depth Profiling at a focused neutron beam to study Li-ion transport in thin-film batteries

ID 45

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In operando observations of Li transport in all-solid-state thin-film batteries during fast (dis)charge cycling, as well as the study of mechanisms of battery aging become possible at a new Neutron Depth Profiling (NDP) setup of JCNS, using the focused neutron beam of reflectometer MARIA (MLZ). This arrangement allows for sufficiently high counting rates necessary for fast, about tens of seconds, measurements under the requirement of a fine, of an order of 10 nm, depth resolution.

## Bringing Neutrons to the User – The HBS Project for Accelerator Based Neutron Sources

ID 48

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With the construction of the high power European Spallation Source (ESS) and the dwindling of reactor based neutron sources in Europe and the US, the neutron user community is facing a mixed outlook towards the availability of neutrons in the coming decades. Accelerator driven neutron sources with high brilliance neutron provision present an alternative to the classical neutron sources to provide scientist with required neutrons to probe structure and dynamics of matter. The Jülich Centre for Neutron Science has started a project to develop and design compact accelerator driven high-brilliance neutron sources as an efficient and cost effective alternative to current low- and medium-flux reactor and spallation sources. Such compact sources will offer access of science and industry to neutrons as medium-flux, but high-brilliance neutron facilities. The “High- Brilliance Neutron Source (HBS)” will consist of a high current proton or deuteron accelerator, a compact neutron production and moderator system and an optimized neutron transport system to provide thermal and cold neutrons with high brilliance. The project will allow construction of a scalable neutron source ranging from university based neutron laboratory to full user facility with open access and service. Embedded within international collaboration with partners from Germany, Europe and Japan the Jülich HBS project will offer flexible solutions to the scientific requirements and establish a new opportunity to exploit neutrons beyond current limitations. We will describe the current status of the project and its partners, the next steps, milestones and the vision for the future neutron landscape in Europe.



## Soft X-ray analysis of microgels and polymer-based microcontainers in aqueous environment

ID 49

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Polymer-based microgel and core-shell particles are versatile containers for the transport of drugs, theranostic gases or catalytically active molecules. They are in many cases biodegradable and fully compatible to in-vivo applications. During recent years we have investigated a broad range of such hybrid materials ranging from gas filled microballoons and thermoresponsive microgel networks to magnetoresponsive particles with embedded iron nanoparticles and core-shell systems filled with theranostic gases or catalytically active fluids (e.g., ionic liquids). Soft X-ray microscopy is an excellent tool to study those systems in-situ in aqueous matrix with high-resolution and chemical sensitivity. We have analyzed shell thicknesses, swelling behaviors and temperature response to contribute better understanding on release mechanisms and have developed procedures to derive high-resolution 3D representations of the particles from 2D transmission X-ray micrographs. The project is funded by the BMBF (05K16WED).



## Incommensurate magnetic systems studied with the three-axis spectrometer MIRA

ID 50

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Incommensurate magnetic structures like Helimagnons and Skyrmions are currently intensively studied. Due to their large size compared to the lattice constant they show excitations at very small  $q$ , where most of the interesting physics is taking place below one meV. The cold-neutron three-axis spectrometer MIRA with its excellent intrinsic  $q$  resolution makes it ideal for studying such excitations in incommensurate magnetic systems. Here we will present several examples for the dynamics of such structures which have been measured with MIRA.

## CSPEC - the cold time-of-flight spectrometer for the ESS

ID 51

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The European Spallation Source (ESS) is expected to be the world's most powerful neutron source. Among the endorsed instruments foreseen for day one instrumentation at ESS, is the cold time-of-flight spectrometer CSPEC, collaboration between the Technische Universität München, and the Laboratoire Léon Brillouin. The high performance cold time-of-flight-spectrometer will serve a broad user community in condensed matter research, life sciences, geosciences or magnetism. 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  $\leq 1.8 \text{ \AA}$  can be probed within each ESS time period. The energy resolution can be tuned in the range of  $\Delta E/E = 6 - 1\%$ , and CSPEC will utilize cold neutrons in the range from  $\lambda = 2 - 20 \text{ \AA}$  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  $\text{mm}^2$  to several  $\text{cm}^2$  in area. The large detector area, with a radius of 3.5 m,  $5 - 140$  degrees and 3.5 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. CSPEC is now in the detailed design phase, and we will present the current status and the expected performance.

## N-doping of Niobium: In-situ EXAFS experiments

ID 52

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*Mr WAGNER, Ralph (Bergische Universität Wuppertal); FRAHM, Ronald (Bergische Universität Wuppertal)*

The preparation of superconducting Nb-cavities for particle accelerators comprises several treatments including e.g. buffered chemical polishing, electropolishing, high pressure rinsing and dry-ice cleaning. Recent experiments have shown that the RF-superconducting properties of the Nb-cavities can substantially be improved using heat treatments under dilute nitrogen or argon atmospheres, yielding a significant reduction of the electrical resistivity. The formation of niobium nitride (NbN) with an increased critical temperature was discussed to explain the observed phenomena, however, a full understanding of the structural changes which lead to these improvements is still lacking. Here we will present results of EXAFS measurements, probing the structural changes during the heat treatments of metallic Nb in-situ. The processing included a treatment in vacuum for 1 h at 900°C and a subsequent exposure to high-purity nitrogen for varying times. EXAFS data were collected prior to any heat treatment as well as during the different process steps at elevated temperature, and after cooling to room temperature. Results of a quantitative EXAFS data evaluation will be presented, showing that the data can be fitted using metallic Nb-Nb coordinations only. No impurity phases such as Nb-nitride were found. In contrast, the disorder parameters for the first few Nb-Nb shells substantially increase with process time and N<sub>2</sub>-exposure. Reasons for such an increased disorder will be discussed.

## Magnetic protein separation with new affinity tags for bare iron oxide nanoparticles

ID 53

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The need for pharmaceuticals based on therapeutic proteins is increasing for many years. Here, the purification is the most cost-intensive step during the production of therapeutic proteins which leads to remarkable potentials in the improvement of common techniques. Besides adaption of purification resin, affinity tags can be used to selectively separate target proteins from contaminations. Commonly, such affinity peptide sequences only bind selectively to expensive surface modifications. Therefore, we design highly affine peptides, utilized as tags for magnetite nanoparticles. Magnetite nanoparticles are synthesized with a co-precipitation route and used for protein purification without further modifications. A glutamate tag is used as affinity sequence in a green fluorescent protein (GFP). High loads of GFP on the magnetite nanoparticles can be reached at different buffer conditions which can be verified by fluorescence analysis. However, the binding of the glutamate tag to the nanoparticles is reversible. The colloidal behavior of the nanoparticles and their interaction with the proteins is investigated with small angle neutron scattering. Here, we are able to observe small aggregates of around 4 primary particles which tend to agglomerate when exposed to proteins and E. coli fermentation broths. However, this agglomeration does not affect the binding capacity while aiding the magnetic separation of nanoparticles. Thus, the developed particle-tag system can be up-scaled.

## Water swelling and exchange kinetics in multi-stimuli responsive PNIPAM-based block copolymer thin films

ID 56

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Multi-stimuli responsive polymers can react with a strong change in volume towards small changes of an external stimulus, which makes them promising materials for a wide variety of applications. [1-3] While the underlying mechanisms of such polymers in solution are well understood, less is known about thermoresponsive polymers in thin film morphology. Regarding this context, the most studied thermoresponsive polymer is poly(N-isopropylacrylamide) (PNIPAM) that exhibits a lower critical solutions temperature (LCST) at 32°. In order to increase the pool of studied thermoresponsive polymers, we focus on the analysis of the swelling and exchange kinetics of a newly synthesized block copolymer. This copolymer consists of a PNIPAM block and a zwitterionic poly(sulfobetaine) (PSB) block that exhibits an upper critical solution temperature (UCST). Time-of-flight neutron reflectometry is used to follow in-situ the swelling and exchange behavior in D<sub>2</sub>O and H<sub>2</sub>O atmosphere. This sophisticated technique enables a large  $qz$  range in combination with a high time resolution of 30 seconds. The kinetic swelling and exchange processes are evaluated with a theoretical model. Detailed insights about the underlying mechanisms are obtained which help to take the next step of implementing such responsive thin films in our daily life.

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[2] W. Wang, et al., *Macromolecules*, 43, 2444 (2010)

[3] A. C. C. Rotzetter, et al., *Adv. Mater.*, 24, 5352 (2012)

## Structure Determination of a new Molecular White-Light Source

ID 57

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The molecular structure of the white light generating amorphous material [(PhSn)<sub>4</sub>S<sub>6</sub>] is investigated using X-ray scattering coupled with a rigid molecular Reverse-Monte-Carlo (RMC) modeling approach. Experimental proof for an adamantane-like molecule structure is found. The intermediate-range structure is analysed, indicating a strong preference for distinct cluster orientations. It is shown that rigid molecular RMC simulations are feasible for structure analysis without using potential-related features. [1-3]

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[3] B. D. Klee, E. Dornsiepen, J. R. Stellohorn, B. Paulus, S. Hosokawa, S. Dehnen, W.-C. Pilgrim, Structure Determination of a new Molecular White-Light Source, submitted to *Physica Status Solidi B* in February 2018.

## Growth and characterization of Fe<sub>3</sub>O<sub>4</sub>/Nb:SrTiO<sub>3</sub> heterostructure

ID 59

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Transition metal oxides are interesting materials to study the complex interaction between electron spin, charge, and orbital ordering. One such oxide is the cubic spinel ferrimagnetic magnetite (Fe<sub>3</sub>O<sub>4</sub>), exhibiting high Curie temperature (83860 K) and a characteristic Verwey transition at 120 K leading to an abrupt increase in its resistivity. The half-metallic ground state (100% spin polarization) with the magnetic moment of 4.05 B/f.u. makes Fe<sub>3</sub>O<sub>4</sub> a promising material for application in spintronics devices. However, for realization of such applications it is important to comprehend these properties in the form of thin film heterostructure on semiconducting substrates [1]. Here we study the morphology, electric, magnetic, magneto-electric coupling and magneto-transport properties of Fe<sub>3</sub>O<sub>4</sub>/Nb:SrTiO<sub>3</sub>. Studies like, charge screening-control of the Verwey transition, interfacial capacitance, magnetic depth profile, and ferroelectric ordering are of special interest. The Fe<sub>3</sub>O<sub>4</sub> films are grown in an oxide molecular beam epitaxy system. We use x-ray diffraction and reflectometry for the structural characterizations, and atomic force microscopy (AFM) for the morphology of thin film. Magnetic and transport properties of the heterostructure are studied using SQUID magnetometer and physical property measurement system, respectively. We use polarized neutron reflectometry technique to study the depth profile of magnetization in the heterostructure.

[1] X. Wang, et. al., J. Mater. Sci. Technol. (in-press) (2018).

## Magnetization and Magnetocaloric Effect in $\text{MnFe}_4\text{Si}_3$

ID 61

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The Magnetocaloric Effect forms the basis of magnetocaloric refrigeration technology, which is a novel energy efficient and environmentally friendly method for cooling [1].  $\text{MnFe}_4\text{Si}_3$  is an interesting magnetocaloric material, which features a modestly large magnetic entropy change as it contains only environmentally unproblematic and abundant elements; it has also some application relevance. Finally the structure contains different transition metal sites, which have recently been discussed as a common structural feature of many transition metal magnetocaloric materials. So a detailed study on magnetization processes is an important prerequisite in understanding this compound. In this contribution we combine results from neutron depolarization experiments [2] carried out using Cryopad setup at single crystal diffractometer POLI at MLZ [3] with direct measurements of the adiabatic temperature change in pulsed magnetic fields using a home-built experimental set-up in HLD [4]. The results will be compared to the ones obtained from the magnetization and heat capacity measurements in static magnetic fields.

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[2] M. T. Rekveldt, Physica B 1999, 267-268, 60-68

[3] V. Hutanu W. Lubertetter, E. Bourgeat-Lami, et.al., Review of Scientific Instruments 87, 105108 (2016)

[4] M. G. Zavareh, C. S. Mejía, A. K. Nayak, et.al., Appl. Phys. Lett. 2015,106.

## Simulation of grazing-incidence small-angle scattering for soft matter studies

ID 62

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Grazing-incidence small-angle scattering (GISAS) is a powerful non-destructive technique to investigate nano- and layered structures deposited to the substrate. Nowadays, GISAS is increasingly used to study various soft matter systems. The disadvantage of GISAS is the challenging data analysis that requires a simulation of the scattering process on a model of the sample. Simulation allows for an understanding of the significance of the various sample properties and accounts for better data analysis and planning of further GISAS experiments. In this contribution we give a short update on the evolution of our software BornAgain [1] and present its application in the recently published studies for analysis of GISAS data for soft matter systems [2-4].

[1] J. Burle, C. Durniak, J. M. Fisher, M. Ganeva, G. Pospelov, W. Van Herck, J. Wuttke, D. Yurov, "BornAgain - Software for simulating and fitting X-ray and neutron small-angle scattering at grazing incidence", Version 1.11.1 (2013-2018), <http://www.bornagainproject.org> (2018)

[2] T. Kyrey, et. al, "Grazing incidence SANS and reflectometry combined with simulation of adsorbed microgel particles" *Physica B* (2018), In Press

[3] H.Frielinghaus, et. al., *Nucl. Instr. and Meth. A* 871, 72-76 (2017)

[4] T. Nylander, et. al., *J. Phys. Chem. B* 121, 2705-2711 (2017)

**POLI: Multifunctional Single Crystal Diffractometer with Polarized Neutrons at MLZ**

ID 70

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POLI is a versatile two axes single crystal diffractometer, mostly dedicated to the investigation of magnetic structures of single crystals using neutron spin polarization.[1] Unlike other shortwavelength polarized neutron diffractometers with Heusler-alloy monochromator for both polarization and monochromatization, POLI employed non-polarized double-focusing monochromators in combination with high-efficiency polarizers. This results in the enhanced intensity of the polarized neutron flux and improved resolution. Also the studies with both polarized and non-polarized neutron diffraction on the same sample under the same conditions (e.g. wavelength, resolution, sample positioning etc.) can be performed. A number of discrete wavelength values from hot (0.29 Å) to near thermal neutrons (1.15 Å) are available by using Cu (220) and Si (311) monochromators on POLI. <sup>3</sup>He spin filter cells are used both to produce and to analyze neutron polarization. This polarization technique is especially efficient for the hot neutrons. For the thermal part of the spectrum, a new dedicated supermirror bender polarizer was developed and is under implementation. POLI is very versatile and flexible instrument which offers different measuring techniques. Namely: (1) polarize neutron diffraction PND (flipping-ratio measurements) using high magnetic field[2], (2) zero-field spherical neutron polarimetry SNP using the third generation Cryopad[3], and (3) classical single crystal neutron diffraction in extreme environments like high magnetic fields, very low/high temperatures, high voltage, pressure cells etc. and their combinations. Rather high flux of hot polarized neutron makes POLI attractive also for the experiments in nuclear physics [4] and even in medical research. In this report, we will show the design of the whole instrument with the emphasis on the polarization components and extreme environments. Several examples of applications in resolving different challenging aspects in crystal, magnetic and domain structure of complex magnetic materials like multiferroics, superconductors, heavy fermion compounds and frustrated systems will be shown.



## Time resolved diffraction of SHS reactions at the HED instrument at European XFEL

ID 71

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Self-propagating high-temperature synthesis (SHS) allows the synthesis of a large variety of compounds and new materials in an energy-efficient way. They have therefore been extensively studied and are well characterized with respect to the equilibrium thermodynamics. In contrast, the characterization of the transient processes before, in, and after the reaction fronts have not been characterized yet due to the extreme reaction rates (propagating with 1 – 150 mm/s), temperatures (up to 4 500 K) and heating rates (between 1 000 and 100 000 K/s). In a project funded by BMBF, our goal is to contribute to the infrastructure for time resolved diffraction experiments in the femtosecond region including an appropriately fast radiometric pyrometry at the High Energy Density Science (HED) instrument at European XFEL. The SHS experiments will be carried out in a small chamber located in the interaction area 2. For the temperature determination, a fast pyrometer built by the HED group of GSI has been adopted. First experiments during commissioning of the instrument will include the formation of silicides, and will focus on structural changes before, in, and after the reaction front. We expect first beam in the beginning of 2019.

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## Neutron reflectivity investigation on thermo-responsive copolymers with embedded magnetic nanoparticles

ID 73

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Nanostructured thin films with embedded magnetic nanoparticles (NPs) are promising in magnetic applications due to their novel chemical and physical properties. The realization of desired magnetic properties depends on the arrangement of the NPs and can be tuned by employing diblock copolymers as host matrixes. Various ordered nanostructured templates can be achieved from the phase separation of diblock copolymers composed of two chemically incompatible blocks. Being coated with one particular type of polymer chains, magnetic NPs can have a selective affinity to one of the blocks and get well distributed within this domain [1]. So far, thermo-responsive copolymers are rarely investigated as host materials for magnetic NPs despite of their high potential. Recently, we investigated the nanostructure and magnetic behavior of hybrid dry films consisting of magnetite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>) NPs coated with polystyrene chains and the diblock copolymer polystyreneblock- polyN-isopropylacrylamide (PS-b-PNIPAM) [1]. Results showed selective incorporation of NPs into the PS domains and a morphological evolution from parallel cylinders to perpendicular ones at low NP concentrations. A superparamagnetic behavior was found irrespective of the NP concentration. Since PS-b-PNIPAM films are most interesting under wet conditions, we extend our investigation from dry films to wet ones in order to explore the swelling behavior of the hybrid films and its impact on the magnetic properties. Neutron reflectivity is used to detect the morphology of hybrid films as a function of temperature in a humid atmosphere. In addition, polarized neutron reflectivity is used to probe the magnetic structure of the PS-b-PNIPAM films with magnetite NPs.

[1] Y. Yao, E. Metwalli, J.-F. Moulin, B. Su, M. Opel, and P. Müller-Buschbaum; ACS Appl. Mater. Interfaces, 6, 18152 (2014).

## Ferrimagnetic films investigated by polarized neutron channeling in planar waveguides

ID 80

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Recently we demonstrated enhanced sensitivity for the investigation of magnetic films with low magnetization about 100 G using a new method, namely polarized neutron channeling in planar waveguides [1]. Such films containing rare-earth elements are promising materials for magnetic recording and switching and nearly inaccessible for the conventional polarized neutron reflectometry because of weak perpendicular magnetization. We use the investigated film as a middle layer in a tri-layer planar waveguide and measure the intensity of neutron microbeam emitted from the exit face of the thin middle layer as a function of the incident grazing angle or the neutron wavelength for spin up and down. The sensitivity of the direct determination of magnetic induction is about 10 G, which is equivalent to about 0.5 permille sensitivity of the magnetization of an Fe material. In [1] we have investigated the magnetically collinear TbCo5 film. In this contribution we report new results of the investigations of the magnetically non-collinear film TbCo11 in non-saturated state. The polarization analysis of the neutron microbeam was used. The results obtained on the fixed wavelength reflectometer NREX (FRM-II, MLZ, Garching, Germany) and the time-of-flight reflectometer REMUR (IBR-2, FLNP JINR, Dubna, Russia) are presented.

[1] S.V. Kozhevnikov, Yu.N. Khaydukov, T. Keller, F. Ott, F. Radu, JETP Lett. 103 (2016) 36.



## Morphology–Function Relationship of Thermoelectric Nanocomposite Films from PEDOT:PSS with Silicon Nanoparticles

ID 88

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Thermoelectric materials pose a compelling technology for power generation from renewable energies, since temperature gradients are transformed into voltages and thus electrical power. So far, highly efficient thermoelectrics comprise rare and/or toxic inorganic materials, and require costand energy-intensive fabrication. These points hinder their large-scale application. In order to overcome these limitations, we pursue a hybrid approach combining the semiconducting polymer blend PEDOT:PSS for its high electrical conductivity and inorganic nanoparticles in order to reduce thermal conductivity within the thin film. Beside the thermoelectric properties, we investigate the thermal conductivity of pristine PEDOT:PSS and of the hybrid film, in order to ultimately calculate the figure-of-merit ZT. Through the introduction of silicon nanoparticles, we found an increase in the figure of merit ZT for intermediate nanoparticle concentrations. Grazingincidence resonant tender x-ray scattering (GIR-TeXS) is used to derive a morphological model, and describe the influence of the nanoparticles on the thermoelectric properties.

## Defect Imaging Using the Positron-Microbeam of the CDB Spectrometer at NEPOMUC

ID 92

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The coincident Doppler broadening (CDB) spectrometer at the positron beam facility NEPOMUC of the research neutron source FRM II enables depth dependent and spatially resolved defect studies by using Doppler broadening spectroscopy (DBS), and element-specific measurements with coincident DBS. In order to investigate the near-surface region and the bulk of a sample, the positron implantation energy can be set up to 30 keV. With a new optional brightness enhancement system, a lateral resolution of 33  $\mu\text{m}$  can be achieved. First measurements on laser beam welds fabricated out of high strength age hardened Al alloys demonstrate the improved performance of the upgraded instrument. The visualization of the defect distribution revealed a sharp transition between the raw material and the welded zone indicating a very small heat affected zone. By coincident DBS the variation of the Cu precipitates and the dissolution of the Cu atoms within the weld could be clearly visualized. Financial support within the project no. 05K16WO7 by the BMBF is gratefully acknowledged.

## Development of a Sample Environment for in-situ Dynamic Light Scattering in Combination with Small Angle Neutron Scattering for the Investigation of Soft Matter at the European Spallation Source ESS

ID 93

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At the moment, with the construction of the European Spallation Source ESS in Lund, the most modern and most powerful scientific neutron source in the world is being built. The aim of our composite project "FlexiProb" is to develop three different sample environments for the investigation of soft matter samples to maximize the potential of the new neutron source with regard to the expected very high neutron flux. These are sample environments for small angle neutron scattering (SANS) combined with in-situ dynamic light scattering (DLS), under grazing incidence (GISANS) and on free-standing liquid films and foams. All sample environments are constructed on an universal carrier system developed at the FZ Jülich to ensure a high repeatability, a maximum flexibility as well as a minimum switching time between different sample environments. The in-situ DLS/SANS module developed in our subproject will provide additional control parameters e.g. the sample stability during the SANS measurements. Thus, we will also develop a special sample holder for about 40 samples which allows the simultaneous measurement of SANS and DLS at two different scattering angles and which provides a precise temperature control. [1]

[1] The authors acknowledge funding from the German Federal Ministry of Education and Research BMBF within the joint project "FlexiProb: Flexible Porobenumgebungen für die Untersuchung weicher Materie an der ESS"(05K2016).



## Temperature Induced Structural Evolution of DMPC-Saponin-Mixtures: From Bicellar to Vesicular Structures?

ID 94

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Saponins are plant derived surfactants which occur e.g. in nuts and exhibit an amphiphilic structure built of a hydrophobic steroidal or triterpenic backbone with a varying number of hydrophilic sugar chains. Some saponins, including aescin, are used specifically for the relief of venous diseases e.g. varicose veins. The interaction of aescin with model membranes composed of the phospholipid 1,2-dimyristoyl-sn-glycero-3-phosphocholine (DMPC) was studied in a wide aescin content range. An incorporation into the lipid bilayer and an aescin domain formation was proven in small, unilamellar vesicles. After membrane saturation with aescin molecules, vesicles get decomposed into very small, bicellar structures. By increasing temperature a conversion back into bigger vesicular structures takes place. Interestingly, these aggregates decompose again when lowering the temperature to about 23°C, the main phase transition temperature ( $T_m$ ) of the lipid DMPC. In the present contribution, the temperature-dependent conversion of bicellar into vesicular structures as a function of the aescin content is investigated with scattering methods. The aim is to elucidate the correlation of the relaxation into the bicellar state and  $T_m$  of DMPC at the molecular length scale. Here, neutron contrast variation is of paramount importance, since it allows to match the contrast of the hydrophobic DMPC membrane part. In this way structural changes in the hydrophilic part of the lipid bilayer can be focused.

## Nanoscience crystallography at a high brilliance laboratory X-ray diffractometer: from mesoscopic to interatomic length scales

ID 95

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The high brilliance laboratory small angle X-ray scattering instrument GALAXI [1] is used to investigate, on mesoscopic length scales, structural correlations in bulk materials or between objects deposited on a surface. The instrument is capable to perform GISAXS experiments in reflection at grazing incidence as well as SAXS experiments in transmission geometry. The X-ray flux on sample is comparable or higher than the one obtained at a comparable beamline at a second-generation synchrotron radiation source. Some results of studies on energy and soft materials as well as materials for information technology will be given. The device properties of thin film heterostructures crucially depend on the structure of the interfaces, not only at the mesoscopic length scale but also at the interatomic length scale. We therefore have the aim to extend the instrument's capabilities towards wide-angle scattering. In this contribution, we will emphasize on the science case of such a development. Also, the possible realisation of this project will be discussed, taking into account the wavelength distribution of the photons emitted by the source.

[1] Jülich Centre for Neutron Science. (2016). GALAXI: Gallium anode low-angle x-ray instrument. Journal of large-scale research facilities, 2, A61. <http://dx.doi.org/10.17815/jlsrf-2-109>

## Theoretical spin-wave dispersions in the antiferromagnetic phase AF1 of MnWO<sub>4</sub> based on the polar atomistic model in P2

ID 96

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Multiferroic properties have attracted much attention since they may have applications in electronic devices. Several models have been proposed to explain the mechanism of magnetoelectric effects such as the change of the modulation wavelength, and another key factor can be a noncollinear spin configuration which is in accord with the theory associated with Aharonov-Casher effect or the inverse Dzyaloshinskii-Moriya interaction. MnWO<sub>4</sub> is an exemplary prototype of magnetoelectric control. At zero field, 3 phase transitions are observed: the commensurate AF1 below 8 K, the incommensurate elliptical spiral spin structure AF2 in 8~12.3 K, and the incommensurate collinear sinusoidal spin structure AF3 below 13.5 K. Its space group has been believed to be P2/c until our studies confirmed the true symmetry P2 and the noncollinear spin-canting structure [1]. With this new magnetic model, it is necessary to re-examine the excitation spectra and the exchange couplings as they are sensitive to the spin configurations. We present spin wave calculations based on the noncollinear magnetic structure; and show good agreements with previous experimental spectra [2]. Interestingly, one of the low-lying excitation modes observed in recent neutron scattering study [3] which cannot be described by the collinear model, may be properly described in this work.

[1] J. Phys. Condens. Matter 30, 135802 (2018)

[2] Phys. Rev. B 83, 140401 (R) (2011)

[3] Phys. Rev. B 93, 214428 (2016)

## Spatially resolved composition and functionality of thin film solar cells

ID 98

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Kesterite  $\text{Cu}_2\text{ZnSnSe}_4$  (CZTSe) and chalcopyrite  $\text{CuIn}_x\text{Ga}_{1-x}\text{Se}_2$  (CIGS) absorber materials can form high efficiency thin film solar cells. They feature a number of different polycrystalline layers with thicknesses of 10 nm up to several  $\mu\text{m}$ . Compositional variations of the absorber layer can limit their conversion efficiency. High resolution, spatially resolved investigations enable insights into such compositional and functional variations by applying X-ray fluorescence (XRF) and X-ray beam induced current (XBIC) measurements, respectively. Simultaneous XRF and XBIC measurements of complete solar cells were conducted in plan-view geometry: The highly focused X-ray beam at the ID16B-NA station of the ESRF scanned the cell and by analyzing the emitted fluorescence radiation and current signal we obtain corresponding maps. As the spatial resolution is about 50 nm, we can show how different grains, grain boundaries and/or different elemental compositions influence the measured current. Compared to other methods, like electron beam induced current (EBIC), complete solar cells can be investigated with high lateral resolution. Combining XRF and XBIC with scanning electron microscopy (SEM) images allows the spatial correlation of energy conversion efficiency and structural/compositional variations of the absorber.

## Printed films of conjugated high-efficiency polymers and small acceptor molecules

ID 100

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Organic Solar Cells are a promising alternative to conventional silicon-based devices as they offer several potential advantages e.g. low weight, high mechanical flexibility and low-cost production. Recent research focuses on identifying new high-efficiency polymers and acceptor molecules to reach high power conversion efficiencies. To date, a Power Conversion Efficiency of 13% could be obtained with a PBDB-T-SF: IT-4F based organic solar cell device. The fluorinated conjugated polymer PBDB-T-SF acts as a donor whereas the fluorinated small molecule IT-4F acts as an electron acceptor. Both molecules are promising for photovoltaic applications as they show higher absorption coefficients, higher efficiency in exciton separation and charge transport as well as enhanced chemical stability as compared with their non-fluorinated counterparts. With a view to commercialization, the solar cell performance must be optimized and an up scale of the thin layer deposition is necessary. Printing of the individual layers of the solar cells can overcome the up-scale challenge. Characterization techniques such as UV/Vis spectroscopy, photoluminescence as well as X ray and neutron scattering methods such as GISAXS/GIWAXS and GISANS/GIWANS can be applied to get a deeper insight into the composition and morphology of the active layer of the printed films with the aim to further improve the solar cell efficiencies.

## Proton exchange membranes for fuel cell applications studied with the Pulsed Low Energy Positron System (PLEPS)

ID 102

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A fuel cell is one of the future promised power sources that produce electricity through an electrochemical reaction of hydrogen and oxygen without producing harmful products. Crosslinking PVA/SSA proton exchange membranes were prepared at different concentrations of SSA (5-30 wt.%) using casting technique. SO<sub>3</sub> groups added by crosslinking with SSA are responsible for modifying the membrane properties as follows: The ionic conductivity of the membranes was enhanced by increasing both temperature and SSA concentration. There is no significant change in the tensile strength of the membranes at a low concentration of SSA (<15 wt.%), above 15 wt.% it decreases significantly. PLEPS operated at the NEutron induced POSitron source MUniCh (NEPOMUC) at FRM II was used to investigate the free volume properties of the prepared membranes. The positron lifetime was measured using a moderated positron having an energy of 16 keV, which provides a mean implantation depth of about 2.8 μm. It is interesting to find that, at low SSA concentration (less than 15 wt.%) the hole volume did not change but increased significantly above 15 wt % SSA. We found that there is a strong correlation between water uptake, proton conductivity and tensile strength with the properties of the free volume.

## Magnetic structures and proton dynamics in phosphatic oxyhydroxides

ID 103

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Geometrically frustrated spin systems comprised of e.g. zig-zag chains, triangles, or pentamers of  $MO_6$  ( $M = 3d$ -metal cations) often show magnetoelectric (ME) coupling effects fundamental in multiferroics. Many phosphatic oxyhydroxides exhibit such interesting moieties of  $MO_6$ . Besides, they contain hydrogen networks of OH- and HOH-groups, showing various strengths of hydrogen bonds, such as  $(Mn^{2+}, Fe^{2+})AlPO_4(OH)2H_2O$ ,  $(Fe^{2+}, Mn^{2+}, Zn)(Fe^{3+})_4(PO_4)_3(OH)_5$ , and  $(Mn^{2+}, Fe^{2+})(PO_4)_2(PO_3OH)_2(H_2O)_4$ . Using elastic/quasielastic scattering studies, their complicate antiferromagnetic structures could be elucidated along with proton tunnelling and super protonic conductivity [1, 2, 3]. These interesting properties and further new findings will be presented at SNI 2018.

1. B. Röska, S.-H. Park, D. Behal, K.-U. Hess, A Günther, G. Benka, Ch. Pfeleiderer, M. Hoelzel, T. Kimura, Determination of the hydrogen-bond network and the ferrimagnetic structure of a rockbridgeite-type compound,  $Fe_2+Fe_3+3.2(Mn^{2+}, Zn)0.8(PO_4)_3(OH)_4.2(HOH)0.8$ , JPCM, accepted on April 27th 2018 (<https://doi.org/10.1088/1361-648X/aac0cd>).
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3. D. Behal, B. Röska, S.-H. Park, B. Pedersen, G. Benka, Ch. Pfeleiderer, Y. Wakabayashi, T. Kimura, The first study of antiferromagnetic eosphorite-childrenite series  $(Mn_{1-x}Fex)AlPO_4(OH)2H_2O$  ( $x = 0.5$ ), J. Magn.& Magn. Mater. 428 (2017) 17-27.

## Restructuring of organic solvents around iron oxide nanoparticles capped by oleic acid

ID 104

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*BRUNNER, Julian (Universität Konstanz); STURM (NÉE ROSEVA), Elena (Universität Konstanz); ZOBEL, Mirijam (Universität Bayreuth)*

Recently, the existence of solvation shells around nanoparticles (NP) was given proof by pair distribution function analysis (PDF) in colloidal suspensions.[1] The building of solvation shells was found to be a universal phenomenon widely independent of capping agent, solvent polarity and particle size. However, a systematic study on how the single parameters influence this restructuring process is still lacking. We performed a synchrotron X-ray study on oleic acid capped iron oxide nanocubes dispersed in toluene, cyclohexane, THF and heptane and corresponding dried powder. The NPs were synthesized via the established thermal decomposition of an iron oleate precursor in solution, which yields highly monodisperse truncated cubic nanocrystals. [2] By subtracting both the solvent background as well as the NP contribution from the PDF of the NP suspension, information on the local molecular ordering of the solvent molecules at the NP interfaces is obtained. Hereby, we detected different restructuring behavior for the investigated organic solvents. A correlation of interfacial ordering and self-assembly behavior of the iron oxide NPs to macroscopic mesocrystals seems at hand. Finally, we will elucidate the formation mechanism and structuration of the mesocrystals, which is not understood so far. [3]

[1] Zobel, M. et al.; Science, 2015, 347, 292

[2] Brunner, J. et al.; Adv. Mater. Interfaces 2017, 4, 1600431

[3] Sturm, E. Cölfen H.; Chem. Soc. Rev., 2016, 45, 5821

## Metal to Nonmetal Phase Separation in Expanded liquid Rubidium

ID 107

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Quantum mechanical calculations of the ground state energy in pure electron gases reveal a mechanical instability, if they are sufficiently expanded. Appropriate scaling allows relating these gas densities to mass densities in real liquid alkali metals which are regarded as perfect model systems for free electron gases. It is found that the region of instability corresponds to 3-4 times the critical density which contradicts the common view of the density induced metal to non-metal transition in these systems located at about twice times the critical density. We have investigated the density dependence of the collective modes in liquid Rb to understand this discrepancy. Collective modes were chosen as suitable probes because their properties are highly sensitive to variations in the interatomic interactions, which are expected to occur if the electron gas ceases to exist. Experiments were carried out on the TOF instruments IN4 and BRISP, at the ILL in Grenoble. We find distinct  $S(Q; \omega)$ -variations when the density range is approached where the electron gas instability is predicted. Variation of mode- and  $S(Q; \omega)$ -maximum properties will be discussed. It is demonstrated that the observed behavior can be understood within a scenario recently suggested for the metal to non-metal transition in liquid Hg where localization of the conduction electrons forces the formation of a micro emulsion consisting of a dense metallic and less dense non-metallic phase. This scenario fully explains the observed  $S(Q; \omega)$  density dependence along the explored density range and throws new light on the mechanism of the density-induced metal to non-metal transition in liquid metals.



## The new Chemical Crystallography Beamline P24 at Petra III (PEX-E), Desy

ID 108

**PAULMANN, Carsten**

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The shutdown of DORIS III discontinued some successful beamlines which served as main stations for a wide field of crystallographic applications ranging from diffuse scattering studies, charge density analysis, phase transitions, disordered and modulated structures all at ambient and nonambient conditions. A collaboration of a joint research BMBF project and Desy build up a new beamline at Petra III dedicated to all fields of crystallographic research. The beamline is in full user operation since spring 2018 and consists of two experimental hutches housing a refurbished Eulerian diffractometer (EH2) and a new heavy-load Kappa-diffractometer (EH2). Both instruments offer a compatible range of detectors (eg. Pilatus 1M CdTe, MarCCD, Ketek drift detector) and sample environments (10K-1300K, electrical fields, DACs). The diffractometers are installed at a standard PETRA III undulator (2m, U29) using a CEMO-type water-cooled DCM (Si111/Si311) with preceding water-cooled mirrors for higher harmonic rejection (Rh/Pt-coated, 1.5 – 3.0 mrad). Due to heat-load restrictions of the CEMO-DCM, additional Cu-coated diamond windows will reduce the low-energy range providing an optimised continuous range between 15keV – 44keV and a smaller range around 8keV. The calculated horizontal and vertical beam profiles at 80m source distance and 17.7keV are 1.5 x 0.7 mm<sup>2</sup> and 0.6 x 0.3 mm<sup>2</sup> FWHM and FW90%M, respectively. Additional Be-CRLs are foreseen for further beam-focusing down to 6µm. The Kappa-diffractometer (ca. 3.5 tons) offers two independent detector circles able to take loads of 30 kg each equipped with motorized counter-weights to balance a detector travel of 500 mm. The sphere of confusion (SOC) of the main circles (incl. Omega) for multi axis movements is below 10 µm and repeatability below 2x10<sup>-4</sup> degrees. The inner circles (Kappa, Phi) provide a SOC below 25 µm for loads up to 5 kg with a maximum load of 10 kg. The Phi circle includes a motorized xyz-stage offering an accuracy in the micrometer range. The available space for sample environments is 190 mm, extendable to 250 mm by removing the stage. In combination with a modular mounting system, an easy exchange of different detectors or sample environments is possible.

## Rotatable load frames for neutron diffraction – analysis of strain, texture, phase transformations and elastic constants

ID 110

**HOELZEL, Markus**

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We present unique load frames which allow an orientation of the load axis by an Eulerian cradle type design and examples of their applications. One version is optimized for texture analysis allowing a free sample rotation around the phi axis under uniaxial tension or compressive stress. Thus complete pole figures under mechanical load can be derived. A second version enables torsion in addition to tension or compression. The load frames were designed at MLZ for the diffractometers SPODI and STRESS-SPEC. However, their compact design allows a usage also on other neutron or synchrotron facilities. In addition to layout and specifications we present selected examples:

- Intensity and strain pole figures in austenitic stainless steels were derived under mechanical load, elucidating the orientation distribution of crystallites as well as lattice strains in one experiment.
- Single-crystalline elastic constants in polycrystalline steels and Ti alloys could be determined under a defined mechanical stress in a method which can be regarded as a reverse of classical stress analysis.
- In monoclinic NiTi shape memory alloys the contributions of domain switching and lattice strains to the macroscopic strain were determined under mechanical load.
- The evolution of martensite variants in polycrystalline NiMnGa under thermo-mechanical load was investigated.



## The new Neutron Depth Profiling instrument at the Prompt Gamma Activation Analysis facility of MLZ

ID 113

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Neutron depth profiling (NDP) uses neutron induced particle emissions to probe, for example, Lithium distributions within the first few micrometers of a sample, yielding valuable insights in fields like battery development. The Prompt Gamma Activation Analysis (PGAA) facility at the Heinz Maier-Leibnitz Zentrum (MLZ) in Garching offers unique opportunities for high-sensitivity NDP by providing neutron fluxes as high as  $4 \times 10^{10} \text{ n / s cm}^2$  with the size of about  $5 \times 5 \text{ mm}^2$ . In the frame of the development of the new NDP instrument, the beam properties, background and resolution limits were investigated thoroughly. The first measurements on suitable samples have been performed. The new NDP instrument together with its installation and the first experiments will be presented with an emphasis on quality assurance. An overview of the future developments in multi-dimensional NDP and NDP combined with PGAA will also be presented.



## Energy research with neutrons (ERWIN) and installation of a neutron powder diffraction option at MLZ

ID 115

*Heere, Michael (KIT & MLZ)*

The need for rapid data collection and studies of small sample volumes in the range of mm<sup>3</sup> are the main driving force for the concept of a high-throughput monochromatic diffraction instrument at the Heinz Maier-Leibnitz Zentrum (MLZ). A large section of reciprocal space will be addressed with sufficient dynamic range and  $\mu\text{s}$  time-resolution while allowing for a variety of complementary sample environments. The medium-resolution neutron powder diffraction (NPD) option for “Energy research with Neutrons” (ErwiN) at the research reactor Munich is foreseen to meet future demand. ErwiN will especially be suited for addressing structural studies and its uniformity of energy-related systems and materials by using simultaneous bulk/spatially resolved NPD. A set of useful experimental options will be implemented enabling time-resolved studies, rapid parametric measurements as a function of external parameters or studies of small samples using an adapted radial collimator. The proposed powder diffraction option ErwiN will bridge the gap in functionality between the high-resolution powder diffractometer SPODI and the time-of-flight diffractometers POWTEX and SAPHIR.

## BATS – a highly flexible option for inverted TOF spectroscopy on the neutron backscattering spectrometer IN16B

ID 118

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Present day reactor based neutron backscattering spectrometers routinely achieve sub-eV energy resolution with Si 111 monochromator and analyser, but are limited in energy transfer range to a window of typically 30 eV. Current scientific applications of this technique from various fields of e.g. energy materials, biology, or glass formers, are dealing with complex systems that demand a broad dynamic range to study processes stretched over multiple time scales. In response to this demand, the recently commissioned BATS option for inverted TOF spectroscopy extends the energy transfer window of IN16B at ILL by a factor of 6. A novel chopper system consisting of two counter rotating disc chopper pairs with multiple slits provide high flexibility of varying the instrumental resolution in seven steps between 1.4 and 9.8 eV for energy windows of 340 eV and momentum transfers up to  $1.9 \text{ \AA}^{-1}$ , with the additional possibility for inelastic offsets. High repetition rate modes with a pulse frequency of 237 Hz are available to trade intensity for energy transfer range, and future use of the available Si 311 analyser allows to reach momentum transfers up to  $3.7 \text{ \AA}^{-1}$  with an expected variable resolution between 6.8 and 59 eV. To improve on a necessary compromise of reducing the chopper slit size below the width of the existing neutron guide, we finally propose a variable focusing and defocusing guide system yielding an expected flux gain of 6 for the smallest slit size.

## Quantitative investigation of the “surface trans-effect” in an adsorbed metal-organic complex by means of X-ray Standing Waves

ID 120

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On-surface coordination chemistry is an emerging field that holds promise for applications in, e.g., nanotechnology, molecular spintronics and heterogeneous catalysis. Previous work provided evidence for a “surface trans-effect”, where the interaction [1] and even the magnetic coupling [2] between the metal ion of adsorbed metal-organic species and the underlying substrate can be affected by ligation of an adduct in trans position. Corresponding changes in the height of the metal center with respect to the substrate were theoretically predicted [3], although they appeared strongly overestimated. Here we present a quantitative analysis of this effect using synchrotronbased normal incidence X-ray standing waves. In particular, for iron phthalocyanine on Ag(111) we determine quantitatively the increase in the height of the Fe ion upon ligation of NH<sub>3</sub> and H<sub>2</sub>O [4]. New dispersion-corrected DFT calculations accurately model this structural effect, and the predicted charge redistribution suggests that the Ag(111) surface acts in a similar manner as a molecular ligand.

[1] W. Hieringer et al., J. Am. Chem. Soc. 133 (2011), 6206.

[2] Ch. Wäckerlin et al., Nat. Commun. 1 (2010), 61.

[3] C. Isvoranu et al., J. Chem. Phys. 134 (2011), 114710.

[4] P. S. Deimel et al., Chem. Sci. 7 (2016), 5647.

## Nuclear resonance scattering polarimetry on single crystals of iron spin crossover compounds

ID 121

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The combination of nuclear forward scattering as time-domain synchrotron-based nuclear resonant scattering method with high-purity polarimetry is a novel approach to probe electronic anisotropies in metal-containing compounds ranging from biological molecules to solid state systems. This method is based on exploring the polarization dependence of the nuclear hyperfine transitions at the 14.4 keV nuclear resonance of  $^{57}\text{Fe}$ . The use of a crossed polarizer-analyzer setup [1] allows the suppression of the incident  $\sigma$ -polarized non-resonant photons. Probing thereby single crystals, the magnitude and orientation of the electric field gradient (EFG) at the Mössbauer nucleus produced by charge anisotropies is deliverable. The application of this method is shown by means of the study of the monoclinic phase of the spin crossover (SCO) complex  $[\text{Fe}(\text{PM-BiA})_2(\text{NCS})_2]$  (1). Iron(II) SCO compounds can be switched reversibly from the low spin to the high spin state, e.g. by variation of temperature [2]. The presence of a strong EFG at the Mössbauer nucleus that leads to a pure electric hyperfine interaction was shown by density functional theory calculations and conventional Mössbauer experiments on a powder sample of 1. The realised nuclear resonance scattering polarimetry experiments on a single crystal of 1 have delivered information in terms of magnitude and orientation of the EFG.

[1] B. Marx et al., Phys. Rev. Letters 110, 254801 (2013)

[2] P. Güttlich et al., Angew. Chem. 106, 2109 (1994)



## Development of new testing machine (load and temperature) for the investigation of super-alloys

ID 122

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Superalloys are widely used for high temperature applications like gas turbines. For this reason these kinds of alloys require specific optimized mechanical properties which they receive from solid solution strengthening, particularly precipitation strengthening. The strength strongly depends on type, shape and size of the respective precipitates. For the improvement of such materials it is crucial to deepen the knowledge of the precipitation kinetics during the whole thermomechanical process, especially during forging and heat treatment. Hence unique sample environment is needed for in situ neutron diffraction and small angle neutron scattering experiments. Here we present the development of a new test rig which can provide tension and compression loading up to 100 kN at elevated temperatures. An attached vacuum furnace allows sample temperatures up to 1200°C. Furthermore the rig will be also used for quenching the sample. First test experiments of the load rig using a VDM Alloy 718 sample during neutron diffraction measurements at the instrument STRESS-SPEC (MLZ) have been performed. After completion of this sample environment as part of a BMBF project (05K16W02), the MLZ will provide this testing machine to the user community. Experiments for the determination of e.g. lattice strain, residual stress and the microstructure in a creep-exposed sample under the influence of temperature will be the focus.



## Structural investigations on PEDOT:PSS polymeric electrodes by means of GISAXS and GIWAXS

ID 123

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One main advantage of organic electronics is the potential of building flexible electronic devices. The polymer mixture poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS) offers good electrical conductivity, transparency and inherent flexibility, which makes it to a promising alternative to rigid and brittle transparent electrodes like indium tin oxide (ITO). By different treatment methods it is possible to adjust the conductivity and work function of PEDOT:PSS to its desired requirements. Conductivities up to 3000 S/cm were achieved, which makes the electrodes competitive to its metal oxide counterparts. The conductivity of PEDOT:PSS films highly depends on the interplay between PEDOT and PSS and its mixing ratio, which has an influence on the morphology and crystallinity. In this work the effect of different treatments on the film structure and conductivity is investigated. Different acid treatments were compared to the commonly used ethylene glycol post-treatment and the pristine PEDOT:PSS thin films. In order to examine the influence of the treatments on the film morphology, crystal structure and orientation, grazing incidence small and wide angle X-ray scattering (GISAXS/GIWAXS) measurements were performed. Taking into account the findings of UV-Vis spectroscopy and 4-point measurements a figure of merit is calculated to link the transparent electrode performance to the structure and crystallite orientation in the PEDOT:PSS thin films.



## The MEPHISTO beam port for nuclear and particle physics

ID 126

*KLENKE, Jens (FRM II)*

The beam port MEPHISTO at the FRM II is dedicated for mostly long term experiments in the field of nuclear and particle physics. The first planned experiment at MEPHISTO is PERC. The author will present an overview of the parameters of the cold white neutron beam and its experimental area in the new east hall. This is based on the already existing neutron optics components, including the neutron guide. The existing additional equipment (as shielding) awaiting the installation at the reactor within the next year.

## Pore filling mechanism in the chemically stable Metal-Organic Framework DUT-67(Zr) by Neutron Powder Diffraction

ID 127

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Metal-Organic Frameworks (MOFs) – hybrid inorganic-organic porous crystalline materials with large and tunable pore structure, surface area and pore volume. The hierarchical pore structure of MOFs and tunable polarity of the inner surface make this class of materials attractive for the application in adsorptive heat pumps. Recently, a chemically stable microporous Zr-based MOF with composition  $Zr_6O_4(OH)_4(tdc)_4(CH_3COO)_4$  (tdc – 2,5-thiophenedicarboxylate) and hierarchical pore structure was synthesized in our group. It possesses a BET area of  $1250\text{ m}^2\text{g}^{-1}$ , total pore volume of  $0.5\text{ cm}^3\text{g}^{-1}$  and shows outstanding stability against water. The material shows desired for the adsorptive heat pump application S-shape water adsorption isotherm and therefore is potentially suitable for adsorptive heat storage applications. In order to shed a light on the mechanism of the water adsorption activated and  $D_2O$ -pre-loaded materials were subjected to the neutron powder diffraction (NPD) study at SPODI instrument of FRM II. Rietveld refinement of the NPD patterns show that the first water molecules are adsorbed at the preferable adsorption sites near the  $\mu_3\text{-O}$  atoms of  $Zr_6O_8$  cluster and trigonal window of the octahedral pore. Further the pore filling proceeds by filling smallest octahedral pore over a middle cuboctahedral and large cuboctahedral pores.

## The beneficial effect of Rubidium in Cu(In,Ga)Se<sub>2</sub> solar cells

ID 129

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Cu(In,Ga)Se<sub>2</sub> solar cells are the most efficient ones among all thin film photovoltaics. The recent push in record efficiencies was mainly realized by applying a RbF post deposition treatment (PDT) to the absorber. However, it is not yet fully clear why the introduced Rb improves the solar cell performance. In order to investigate the beneficial effect of Rb, a Cu(In,Ga)Se<sub>2</sub> absorber was grown on a Mo coated alkali free substrate and subjected to a RbF PDT. This pure RbF PDT leads to a significantly higher conversion efficiency. A thin cross sectional lamella was cut out of the layer stack and investigated via a combination of different electron microscopy techniques and synchrotron based X-ray fluorescence analysis. It is evident that Rb segregates at random grain boundaries and dislocation cores, where it likely passivates defects. In contrast, Rb does not segregate at benign  $\Sigma 3$  twin boundaries. Additionally, Rb agglomerates at the interface between the absorber and the MoSe<sub>2</sub> layer. Our results thus provide clear indications of the origin of the beneficial effect of Rb in Cu(In,Ga)Se<sub>2</sub> solar cells [Schöppe et al., Nano Energy 42 (2017) 307]. Subsequently, we investigated a high efficiency solar cell grown on a glass substrate and subjected to a RbF PDT, thus providing a conversion efficiency of over 20 %. Applying the same combination of analysis techniques, we clearly demonstrate that our conclusions for a pure RbF PDT are also valid for state of the art devices.

## Nanometer and sub-nanometer layer reference samples for X-ray fluorescence - New results and applications

ID 131

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Non-destructive X-ray spectrometry methods such as XRF, TXRF as well as GIXRF/GEXRF allow determining the elemental composition of unknown bulk and layered samples. However, in most cases well-defined standard or reference samples are required if quantitative information about layer thickness (nm) or mass deposition ( $\text{ng}/\text{mm}^2$ ) shall be deduced from the fluorescence radiation (cps). Internal or external standards such as dried or  $\mu\text{L}/\text{nL}$ -droplets may be used for this quantification but problems can occur in sample preparation, measurement and evaluation. XRF signals from the sample carrier may further increase the signal background and decrease sensitivity especially for low concentrations. Ultrathin silicon nitride membranes have been used as low-background reference sample substrates for several years. Further, dedicated physical vapor deposition (PVD) methods are applied to provide laterally extremely homogeneous layered multi-element reference samples of welldefined mass deposition in the range of few nm and even much below the mass deposition of an atomic monolayer. As fabrication and characterization becomes cost-efficient for larger production batches, market research and evaluation of demand in research and industry is essential. New reference systems are suggested, focusing on transition metals of period 4 (3 to 8 keV) or period 5 and 6 (8 to 21 keV), plus multi-element sub-monolayers in the pm range.

## Near-surface region characterization of nitrogen treated single-crystal Nb (100)

ID 133

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Modern particle accelerators rely on niobium RF (Radio-Frequency) cavities for their operation and there is a big drive for performance improvement of such devices. Achieving a higher quality factor (Q0), will lead to higher luminosity while reducing the dynamic heat load, resulting in potential cost savings. Nitrogen doping is known to increase the performance of niobium cavities [1], however, the physical and chemical processes and phenomena involved are not yet understood [1,2]. In this work, the niobium (100) surface was subjected to a step-wise preparation based on the so-called 'nitrogen infusion' process, which has recently showed an increase in Q0 and accelerating gradient for 1.3 GHz SRF cavities [3]. The progressive dissolution of the natural oxide layers upon temperature increase and nitrogen presence were tracked in-situ by means of X-Ray Reflectivity (XRR) while the effects of oxygen and nitrogen interstitials was retrieved from Depth-Resolved Grazing Incidence High Energy Diffuse X-Ray Scattering (GIXRD) measurements obtained at ESRF beamline ID31. Additional surface sensitive characterization techniques, namely X-Ray Photoemission Spectroscopy (XPS) and Scanning Electron Microscopy (SEM) were performed at DESY Nanolab to bring further understanding to the system.

[1] A. Grassellino et al, Supercond. Sci. Technol.26 102001(2013).

[2] P. Dhakal et al, IEEE Tran. on App. Superc.25 3500104(2015).

[3] A Grassellino et al, Supercond. Sci. Technol.30 094004(2017).



## Neutron Optics from NOB Nano Optics Berlin

ID 134

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Together with the Helmholtz-Zentrum Berlin NOB has developed several kinds of neutron optical devices for polarization, collimation and focusing of neutron beams. In most of them the neutrons are transported in thin silicon wafers with coated walls. Results are shown for solid state benders and a radial bender for the polarization of neutrons. A solid state polarizing bender without absorbing layers used together with a collimator allows polarizing or analyzing neutrons without deflecting them. Two-dimensional polarization analysers for an angular range of 5 degrees in both directions are presented. All these devices have polarizations of 95%. In the last years 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 with a polarization above 98% and a maximum transmission above 65% [1]. Further S-benders for shorter wavelengths were built and tested as well. A focusing solid state lens was made from Si wafers coated with m=2 Ni-Ti supermirrors. Here a focus with a FWHM of 2.4 mm was reached and an intensity increase of 5.6 compared to the intensity without the lens

[1] Th. Krist, F. Rucker, G. Brandl, R. Georgii: High performance, large cross section S-bender for neutron polarization, Nuclear Inst. and Methods in Physics Research, A 698 (2013) 94-97.



## P65 - a workhorse beamline for in-situ and operando XAFS spectroscopy at the PETRA III storage ring

ID 136

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X-ray absorption fine structure spectroscopy (XAFS) is among the most useful analytical methods for the investigation of structure/function relations in all fields of catalytic chemistry and energy storage techniques like batteries, fuel cells, H<sub>2</sub> storage etc. The beamline was built as part of the PETRA III extension project and is in regular user operation since Summer 2016. The beamline was designed to provide stable and reliable operating conditions for XAFS in bulk samples to employ XAFS as analytical method in applied science. A short undulator provides a monochromatic photon flux of up to  $10^{12}$  s<sup>-1</sup>, more than enough for all kinds of XAFS experiments. The radiation from the undulator is monochromatised with a water cooled double crystal monochromator. A pair of plane mirrors is employed to reduce the contamination of the monochromatic beam by higher harmonics radiation and to reduce the heat load on the first DCM crystal. A typical EXAFS scan in transmission mode takes 60 s – 180 s. The spot size on the sample is  $0.5 \times 1$  mm<sup>2</sup>, a size that fits very well to catalytic in-situ/operando samples. The experiment is equipped with supply and safety infrastructure for all kinds of gases that are used in in-situ experiments. A small sample preparation lab is available directly beneath the beamline. We will present the beamline design and demonstrate the performance and versatility of the beamline using selected examples from experiments done during the first 2 years of operation.

**FIREPOD – the fine resolution powder diffractometer @ Berlin research reactor BER II**

ID 137

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The fine-resolution neutron powder diffractometer E9 [1] at the BER II research reactor at the Helmholtz-Zentrum Berlin für Materialien und Energie is dedicated to collect high quality diffractograms suited for crystal structure determinations and Rietveld refinements. The detector bank consists of eight individual 2D detectors, arranged at an optimized, non-constant distance from the sample, and a radial collimator to reduce background noise. Position-sensitive data integration results in a strongly reduced peak asymmetry. Through the choice of sample diameter, axial focus length, and primary collimation a wide range of combinations of intensities, resolution curves, and sample volume can be obtained. The instrument allows the use of a large variety of sample environments, covering temperatures from 1.5 - 2000 K, pressure up to 2.5 kbar, variable magnetic fields (up to 5T) and 10 different gasadsorption modules (4 K to 1500 K and up to 10000 bar). Possible load gasses include nitrogen, hydrogen, heavy hydrogen, argon and helium. For room temperature measurements we offer a 10-fold automatic sample changer.

[1] Helmholtz-Zentrum Berlin für Materialien und Energie. (2017). E9: The Fine Resolution Powder Diffractometer (FIREPOD) at BERII. Journal of large-scale research facilities, 3, A103



## High-Flux XAFS-Beamline P64 at PETRA III

ID 139

**CALIEBE, Wolfgang (DESY)**

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Beamline P64 is dedicated to XAFS-experiments which require high flux like QEXAFS on the 10ms time-scale, EXAFS of highly diluted systems, and resonant high-resolution x-ray emission spectroscopy. We will present the optical set-up of the beamline (undulator, monochromators, mirrors, detectors), characteristics of these components, describe different operation modes for EXAFSscans (continuous versus step-scans), and show some typical data from the first 2 years of user operation. The time required for one energy scan varies from 10ms (QEXAFS) to 1200s (measurements of highly diluted systems). In the first case, the undulator is tapered in order to broaden its energy spectrum, and a special monochromator oscillates with a frequency of 50Hz while the intensities of the detectors and the angle of the monochromator are measured simultaneously with 2MHz. In the latter case, monochromator and undulator move together from one energy-point to the next, and the intensities are recorded (step-scan). This type of scan is applied for highly diluted samples in combination with a 100-element energy-dispersive Ge-Detector. Conventional continuous scans take 60-300s. So far, users have performed experiments in the fields of solid state physics, catalysis, bio-chemistry, biology, and environmental sciences with different sample environments like cryostats, electrochemical cells, capillary-reactors, with liquid jets, or as conventional pellets in air.

## Beamline P02.1: A Workhorse for High-Resolution Powder Diffraction & Total Scattering Experiments at PETRA III, DESY

ID 143

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Powder diffraction is a standard tool for characterisation in a wide range of fields across the physical sciences: from materials science and metallurgy, to chemistry and solid state physics. Third generation synchrotron sources provide high fluxes of X-rays at higher energy than laboratory sources. These characteristics open the door not only to rapid data collection for high spatial resolution structural studies, but also to time-resolved in situ/in operando measurements of functional materials under real conditions, thanks to high energy X-rays being able to penetrate operating environments. Furthermore, the use of high energy X-rays increases the region of reciprocal space which may be studied in an experiment, facilitating improved spatial resolution in total scattering measurements. Beamline P02.1 (PETRA III, DESY, Hamburg, Germany) is almost unique amongst high-resolution powder diffraction instruments in operating at a fixed energy of 60 keV [1],[2]. This energy is particularly well suited to the collection of powder diffraction data for Rietveld refinement (especially for high-Z materials, where absorption is a problem at lower energy) and for collection of total scattering data (the high energy facilitating QMax of at least 20 Å<sup>-1</sup>), which may be interpreted by Pair Distribution Function (PDF) analysis. Currently two detectors are available on the beamline (a two-dimensional Perkin Elmer XRD1621 and an in-house developed 10-channel Multi-Analyser Detector), whilst additional devices are available from a shared detector pool (including a Dectris Pilatus3 CdTe 2M designed for high-energy data collection). In addition to ambient temperature capillary measurements, P02.1 offers a range of standard sample environments (e.g. hot-air blower – RT-1100 K; cryostream – 90-500 K; cryostat – 10-300 K) whilst a number of others are under development (e.g. low pressure gas adsorption cell) or are maintained by the PETRA III sample environment group. Recently we have also brought a sample changing robot into operation, with capacity for 300 samples (see figure); this device is compatible with the cryostream and hot-air blower environments. Furthermore, the enormous amount of free space around the diffractometer, compared to many other powder diffraction beamlines, allows easy integration of complicated sample environments which are brought to the beamline by our users. We report here the latest developments at P02.1 including planned changes to detector configurations, new sample environments, updates to the control software and possible remote access schemes.

## Heterogeneous local order in self-assembled nanoparticle films revealed by X-ray cross-correlations

ID 149

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Accessing structure beyond pair correlation functions is expected to shed light on various open questions in condensed matter physics such as the glass transition phenomenon. Typically, it is governed by the appearance of dynamical heterogeneities, suggested to be closely connected to spatial heterogeneities. Such spatial motifs are believed to play a key role in the glass transition process, e.g., via geometrical frustration suppressing formation and growth of crystallites. In order to measure such structural heterogeneities, higher-order correlation functions have to be defined using e.g. the X-ray Cross Correlation Analysis (XCCA) technique [1,2]. Here, we will discuss our recent XCCA results on colloidal films and crystals [3,4]. A special focus is set on the self-assembly of functionalized gold nanoparticles [5]. Depending on the initial concentration of particles, structurally heterogeneous films are formed with dominating four- and six-folds symmetry. The amplitude of order parameters indicates that a minimum sample amount is necessary to obtain well-ordered structures. This richness of information cannot be achieved by standard microscopy techniques that are commonly used to characterize such nanoscale systems.

[1] P. Wochner et al. PNAS 106, 11511 (2009).

[2] F. Lehmkuhler et al. J. Appl. Cryst. 47, 1315 (2014).

[3] M.A. Schroer et al. Soft Matter 11, 5465 (2015).

[4] F. Lehmkuhler et al. J. Appl. Cryst. 49, 2046 (2017).

[5] F. Lehmkuhler et al. IUCrJ 5, 354 (2018).



## Zone-plate based soft x-ray microscopy with sub-10 nm resolution

ID 150

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Soft x-ray scanning transmission microspectroscopy (STXM) using Fresnel zone plates (FZPs) as focusing elements has developed into a routine technique for the investigation of semi-transparent thin film specimens. The x-ray absorption contrast is utilized as elemental or chemical fingerprint in multinary component specimens. Routine operation of STXMs uses spatial resolution of around 30 nm determined by the outermost zone width of the FZP. An elegant method to fabricate highresolution FZPs has been introduced by doubling the line density obtained from the lithography step utilizing atomic layer deposition (ALD). We have recently prepared FZPs with line structures to about 7 nm thus pushing the resolution limits into the sub-10 nm regime. We performed the experiments at the SLS PoLux-STXM and the HERMES beamline at SOLEIL to evaluate the influences of energy resolution, coherence length and FZP parameters on the achievable resolution. In order to obtain ultimate resolution, interferometric positional feedback is required. We will report on the resolution tests of specific test samples but also on technologically relevant specimens where the improvement in lateral resolution becomes inevitable. Determination of the spatial resolution was conducted using a two-dimensional Fourier shell correlation of two independent data sets. This yielded a frequency cut-off at  $0.15 \text{ nm}^{-1}$  in Fourier space, or 6.8 nm (7.1 nm) in real space considering the half-bit (one bit) criterion.

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## Critical scattering in classical and nearly quantum critical systems

ID 154

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We report on a study of critical scattering in classical and nearly quantum critical antiferromagnets (AFs). The energy width of the critical scattering was determined by high-resolution neutron spin echo at TRISP at the MLZ. The classical systems studied include the  $s=5/2$  AFs  $\text{MnF}_2$  and  $\text{Rb}_2\text{MnF}_4$  with quasi 2D and 3D spin interactions, respectively. Discrepancies between experiment and theory observed in previous three-axis studies could be resolved by our high-resolution measurement. For a study of quantum critical systems, we chose the  $\text{Ce Cu}_{6-x}\text{Au}_x$  series, which has a quantum critical point at  $x=0.1$  and shows AF ordering for larger  $x$ . First measurements at  $\text{Ce Cu}_{5.8}\text{Au}_{0.2}$  ( $T_N=0.22\text{K}$ ) show a hitherto unexplained dynamical critical exponent.

## Modeling of small-angle scattering data from proteins with modifications

ID 155

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For the analysis of solution small-angle scattering data of proteins a wide selection of software such as Crysol [1] exists. In the strict sense they are only designed for pure protein structures. Many proteins, however, come with modifications such as fatty acids, glycosyl groups [2] and bile acids [3]. In a few cases such modifications could be accurately modeled together with their underlying protein structure. E.g. for PEGylated hemoglobin, where the electron density of PEG and hemoglobin are very similar, a bead model for the protein and its modification could be successfully applied [4]. An analytical description of the scattering of the PEG chains (Gaussian chain model) worked well, too [5]. However, modifications often differ significantly from proteins with regard to their electron density and hydrophobicity and cannot always be modeled analytically. To account for the different specific density between the modification and the protein we implemented a model in the freely available “Will It Fit?” framework [6] which allows using different scaling factors for the displaced volumes of the modification- and protein-related atoms. The poster will give an overview on the modeling of proteins with modifications and show first results on solution SAXS data.

[1] J.Appl.Cryst. 28 768 (1995)

[2] Biophys.J. 96 153 (2009)

[3] Biochem. 43 5987 (2004)

[4] Biophys.J. 94 173 (2008)

[5] Langmuir 2015 31 8402 (2015)

[6] J.Appl.Cryst. 46 1894 (2013)

## Operando surface X-ray diffraction studies of electrodeposited Co oxide thin films during oxygen evolution

ID 157

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Water splitting is one of the most promising routes for renewable energy conversion and storage. Iron group metal oxides, especially Co oxides, are promising candidates for noble metal free catalysts for the oxygen evolution reaction (OER). In order to design efficient and scalable catalyst materials the relation between the surface structure and the catalytic properties are of fundamental importance. In this work we performed operando studies of electrodeposited Co<sub>3</sub>O<sub>4</sub>/Au(111) and CoOOH/Au(111) thin films during oxygen evolution using surface X-ray diffraction (SXRD). The SXRD measurements were done in our novel operando XRD cell which allows measurements during gas evolution and current densities of up to 30 mA/cm<sup>2</sup>. As samples thin epitaxial (111) oriented Co<sub>3</sub>O<sub>4</sub> and (001) oriented CoOOH films were used. SXRD measurements during potential cycles into the OER regime reveal a clear reversible decrease in film thickness of about 1 nm for the Co<sub>3</sub>O<sub>4</sub> film between 0.3 V and 0.6 V whereas no structural changes were observed in the case of CoOOH. The onset potential of OER is the same for both films which suggests that the topmost layer of Co<sub>3</sub>O<sub>4</sub> is converted to CoOOH and the active phase for the OER is the same in both cases. Since the CoOOH thickness is constant (within 0.05 nm), only changes in the surface termination of the catalyst seem to occur in the OER regime.

## New setup for in-situ MBE growth studies using high energy x-ray diffraction

ID 162

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In the past, the structure of thin epitaxial films has been studied extensively by hard x-rays using dedicated systems at various synchrotron beamlines for in-situ studies [1,2]. The typical time scale for these experiments has been rather high forcing these studies to focus either on a rather limited parts of reciprocal space [3] or to accept rather poor time resolution. Using high energy x-rays, however, surface diffraction is able to record huge maps of reciprocal space during catalytic reactions within a few seconds [4]. Here, we present a new setup to extend high energy x-ray diffraction (HEXRD) to in-situ studies of thin epitaxial film during molecular beam epitaxy (MBE) with high time resolution while large parts of reciprocal space are recorded. Thus, real time studies during MBE are possible. The deposition chamber is mounted on the surface diffractometer at the high energy material science beamline P07 at PETRA III. During the experiment, the sample is moved by the diffractometer while the chamber is fixed. In addition to structural investigations, a hemispherical electron spectrometer is implemented for complementary x-ray photoelectron spectroscopy (XPS) measurements.

[1] S. Ferrer et al., Rev. Sci.Instr. 66, 1674 (1995)

[2] C. Nicklin et al., J. Synch. Rad. 23, 1245 (2016)

[3] O. Kuschel et al., Appl. Phys. Lett. 111, 041902 (2017)

[4] J. Gustafson et al. Science 343, 758 (2014)



## In-situ Microspectrophotometry for Macromolecular Crystallography

ID 163

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Many proteins contain a chromophore or metal centre with absorption bands in the UV/VIS spectral region. The details of the absorption bands report on the state of the protein, like different oxidation or conformation states. In X-ray crystallography, the absorption may even reflect radiation damage during data collection. Especially in high-resolution data collection, alterations in the protein structures are still a severe problem. In-situ microspectrophotometry is a valuable tool to monitor alterations in the UV/VIS absorption of a protein and enables the characterisation of a state of a protein during data collection. In the HZB MX-group a setup for an in-situ UV/VIS microspectrophotometry was build up recently and will be installed for on-line experiments. It consists of a focussing optics directly mounted on the sample stage of the diffractometer. Optical fibres are used to illuminate the protein crystal from a UV/VIS light source and to connect to a compact spectrometer for data recording.



## Upgrade of the NEPOMUC remoderator

ID 164

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The neutron-induced positron source Munich NEPOMUC provides a monochromatic positron beam of  $> 10^9$  e<sup>+</sup>/s and  $\approx 10$  mm diameter FWHM. To create a small beam focus or sharp pulses of  $\approx 100$  ps the beam brightness needs to be enhanced by remoderation. This is achieved by focusing the beam magnetically on a tungsten single crystal W(100). Afterwards the beam exhibits an intensity of  $> 3 \cdot 10^7$  e<sup>+</sup>/s and a diameter of  $< 2$  mm FWHM. However, we can further optimize the quality and increase the intensity of the remoderated beam by systematic tests of different remoderator materials and a precise positioning of the remoderator within the focus of the magnetic lens. Therefore, a new remoderation unit has been designed to allow a replacement of the remoderator crystal within several minutes. In addition, it will be possible to heat the crystal and to treat its surface with different gasses, e.g. atomic hydrogen. Moreover, in-situ cooling with liquid Helium will offer the opportunity to use noble gasses like Ne as remoderators. After the upgrade we expect a higher remoderation efficiency and an increased brightness with higher beam intensity that is valuable for applications like the Coincidence Doppler-Broadening Spectrometer or the Scanning Positron Microscope. Moreover, a high intensity will further reduce the measurement time.



## New possibilities for Hard X-Ray Photoelectron Spectroscopy at DESY

ID 165

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A new X-ray undulator beamline dedicated to HAXPES applications is now open for user operation at PETRA III (DESY, Hamburg). The beamline comprehends a unique selection of HAXPES techniques using specialized instruments built and operated in collaboration with external user groups. The main instrument is the established HAXPES setup relocated from PETRA III beamline P09. It provides an optional wide-angle lens for increased transmission and/or angle resolved studies as well as an add-on spin selective detector employing an improved 2D spin filter which is currently being commissioned. The HAXPEEM instrument for spectromicroscopy applications utilizing the depth sensitivity in the keV energy range. A third instrument facilitates in-operando studies of catalytic reactions at industrially relevant pressures up to 10 bar. As a further development, a novel instrument combining full-field k-microscopy with time-of-flight (ToF) parallel energy recording will be tested at the beamline to measure the 4D spectral function  $\rho(E, B, k)$  in the HAXPES regime. The X-ray source is a 2m X-ray undulator covering an energy range from 2.4 to 30 keV while wave length selection is facilitated by LN<sub>2</sub>-cooled double Si-crystal monochromator with interchangeable Si(111) and (311) crystal pairs. Furthermore, a 4-bounce post-monochromator and a diamond phase plate are available. The expected minimum spot size at the first instrument position is  $\sim 10 \times 10 \mu\text{m}^2$  providing about  $2 \times 10^{13}$  ph/s (Si(111) at 4-6 keV).

## Sample environment and Applications of the Hot Neutron Single Crystal Diffractometer HEiDi@MLZ

ID 166

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The single crystal diffractometer HEiDi is designed for detailed structural studies related to physics, chemistry and mineralogy by offering high flux, high resolution and large  $q$  range, low absorption and high sensitivity for light elements using hot neutrons from FRM II at the Heinz Maier-Leibnitz Zentrum (MLZ). In order to fulfil the various needs of our users the instrument offers a broad suite of sample environments, for instance low temperatures down to 2.5 K for studies on quantum phenomena like complex magnetic behaviour in multiferroics or superconductivity in Cu- or Fe-based compounds, and high temperatures up to 1000°C for studies on ionic conductors using an in-house (MLZ) developed mirror furnace with tunable atmospheres [F. Magro Sastre; master thesis, 2017]. Recently, a BMBF funded project (BMBF 05K16PA) on small samples and high pressure cells has been launched to add high pressure studies up to 5 GPa to the available options on HEiDi (for details, please have a look on A. Grzechniks presentation on this conference and e.g. A. Grzechnik et al; J. Appl. Cryst. 51, 2018). Aside from this, some scientific examples like the oxygen diffusion pathway in  $(\text{Nd/Pr})_2\text{NiO}_{4+\delta}$  [e.g. M. Ceretti et al; Inorg. Chem. 57, 2018] and studies on the hydrogen bonds with different degrees of disorder in different silicate and phosphate-based gems [e.g. D.G. Gatta et al; Phys. and Chem. of Min., 2018] will be presented in order to underline the versatile applications of HEiDi, e.g. in materials for energy research or earth sciences.

## The interaction of viral fusion peptides with model lipid membranes at high hydrostatic pressure

ID 168

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When a virus enters a host cell, the insertion of viral fusion peptides (FPs) into the target membrane catalyzes the membrane fusion reaction. While fusion intermediates with high membrane curvature appear in this process, the exact mechanisms of the peptide/membrane interactions remain unclear up to now. We investigated the insertion mechanism of different FPs into model membranes in X-ray reflectivity measurements at the interface between monoolein/water mixtures and a silicon substrate. In addition, the bulk and interfacial structures were investigated with small angle X-ray scattering in transmission and in surface-sensitive grazing incidence. Monoolein/water mixtures have a very rich phase diagram, which can be tuned by pressure. Notably, the inverse bicontinuous cubic phases exhibit structural analogy to the hemifusion intermediates. Previous studies demonstrated the effect of FPs on the pressure-dependent phase boundaries [1]. We found that pressurization triggers formation of ordered lamellar monoolein multilayers at the hydrophilic surface even in a pressure range where the bulk material is in the cubic phase. We resolved the vertical membrane structure of these multilayers and monitored closely the penetration of FPs into the membrane. Experiments were performed in a custom-made high hydrostatic pressure cell [2] at beamlines ID31 of the ESRF and BL9 of DELTA.

[1] A. Levin et al, J Phys Chem B 121 8492-8502, 2017

[2] F.J. Wirkert et al, J. Synchr. Radiat. 21 76-81, 2014

## The new high-resolution neutron spin-echo spectrometer at MLZ: J-NSE Phoenix

ID 169

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The neutron spin echo spectrometer provides the ultimate energy resolution in quasi-elastic cold neutron scattering spectroscopy. High-resolution means the extension of Fourier-time ( $\tau$ ) up to the regime of  $\mu\text{s}$ , which depends on the reduction of the field-integral inhomogeneity down to the some ppm. The limiting factor is the performance of the present correction coils. We could mitigate this hardship by optimizing the shape of the precession magnetic field - and thus reducing the intrinsic inhomogeneity introduced by the cylindrical main solenoids - guided by the original idea of Zeyen [1] and by a new semi-analytical approach [2]. Following this strategy we numerically optimized and realized a set of new superconducting, fringe-field compensated solenoids with zero net dipole moment for the neutron spin-echo spectrometer J-NSE at MLZ. Due to the lower intrinsic field inhomogeneity the amount of required correction is reduced by over a factor 2, as expected from numerical calculations. After the installation of the new precession coils the performance of the new J-NSE Phoenix already allowed us to use a magnetic field integral of 1.0 Tm for a Fourier-time of 100 ns at 8 Å, 190 ns at 10 Å and 350 ns at 12.5 Å. Together with an increased wavelength-band from 10% to 20% we will strive for 500 ns at 15 Å. The improved resolution may be used to reach larger Fourier-times and/or to benefit from significant intensity gains by the use of shorter neutron wavelength from a given  $\tau$ . Thus the new J-NSE will meet the needs to look into the microscopic dynamics of functional polymers thanks to new and more enhanced qualities.

[1] Zeyen C.M.E. and Rem P.C., 1996 Meas. Sci. Technol. 7 782–91,

[2] Pasini S. and Monkenbusch M., 2015 Meas. Sci. Technol. 26 035501

## High-temperature stability of matrix in boron-containing Co-Re-Cr alloys for gas turbine applications

ID 170

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Co-Re-based alloys [1] are developed to supplement single crystal Ni-based superalloys in future gas turbines. Addition of boron to the Co-Re-based alloys largely increases their ductility [2] and is thus advantageous. On the other hand, the stability of the matrix at the foreseen metal operation temperatures ( $\geq 1200^\circ\text{C}$ ), which can be influenced by the presence of boron, is also an important concern for the alloy development. Therefore, the influence of boron addition on Co-Re stability was investigated in situ at high temperatures using neutron diffraction (MLZ Garching). It was found for the Co-17Re-23Cr alloy that the increased boron concentration changes significantly, but not monotonically, the hcp to fcc matrix transformation temperature [3]. The reason is an interplay between amount of boron in the matrix and amount of sigma phase ( $\text{Cr}_2\text{Re}_3$ ) which binds hcp-stabilizing element Re. Moreover, increasing boron concentration also lowers temperature at which evaporation of Co and/or Cr occurs, followed by an appearance of second or even third hcp matrix phase. The formation of these additional hcp phases was observed at temperatures  $> 1430^\circ\text{C}$ . This effect is not detrimental to the alloy development as the evaporation starts significantly above the foreseen operation temperature.

[1] J. Rösler et al., Adv.Eng.Mater. 9, 2007, 876

[2] D. Mukherji et al., Scr.Mater.66, 2012, 60

[3] P. Strunz et al., Met.Mater.Int., 2018, doi.org/10.1007/s12540-018-0121-8

## Investigating the interaction of water with vanadium oxide surfaces by XPS

ID 172

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The adsorption of molecules on vanadium oxide surfaces has been studied extensively, due to their widespread use in industrial catalysis. X-ray photoelectron spectroscopy (XPS) provides detailed information on the chemical bonding states of adsorbed molecules, and how they influence surface properties. However, previously existing XPS studies of molecules on vanadium oxide surfaces were limited to those performed in high vacuum, and thus may not represent the conditions present in industrial reactions. Here, we bridge this “pressure gap” with “ambient pressure” XPS, where we have used synchrotron and lab X-ray sources to study vanadium oxide surfaces in the presence of water at pressures up to 2 Torr. These measurements provide insights on how moisture affects the surfaces of various oxidation states (V<sup>3+</sup>, V<sup>4+</sup> and V<sup>5+</sup>) at catalytically relevant temperatures up to 400°C. We also explore the behaviour of the system across the metal-insulator transition in VO<sub>2</sub>.

## Low-temperature phase transitions in PrAlO<sub>3</sub>–SrTiO<sub>3</sub> series

ID 173

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Phase and structural behaviour of the continuous perovskite solid solution Pr<sub>1-x</sub>Sr<sub>x</sub>Al<sub>1-x</sub>Ti<sub>x</sub>O<sub>3</sub> have been studied in the temperature range 20 – 295 K by high-resolution X-ray synchrotron powder diffraction. Superb characteristics of the beamline ID22@ESRF allows to detect either subtle splitting of the main perovskite maxima and/or appearance of weak superstructure reflections, thus proving diverse variants of perovskite structure existing in Pr<sub>1-x</sub>Sr<sub>x</sub>Al<sub>1-x</sub>Ti<sub>x</sub>O<sub>3</sub> series at different compositions and temperatures. It was revealed that the samples with  $x = 0.1$  and  $0.2$  undergo a sequence of structural phase transitions R-3c – Immb – I2/m, similar to those observed for the parent PrAlO<sub>3</sub> phase. These LT transitions in praseodymium aluminate are unique among all RAlO<sub>3</sub> perovskites and are considered to be caused by the electronic effects involving Pr<sup>3+</sup> ions, e.g. a coupling between Pr<sup>3+</sup> electronic states and phonons and/or cooperative Jahn-Teller effects. It was established that temperatures of both R-3c – Immb and Immb – I2/m transitions in Pr<sub>1-x</sub>Sr<sub>x</sub>Al<sub>1-x</sub>Ti<sub>x</sub>O<sub>3</sub> series systematically decrease from 205 K and 151 K for PrAlO<sub>3</sub> to 170 K and 90 K for  $x = 0.2$  sample. Quite different phase behaviour was observed in the SrTiO<sub>3</sub>-rich part of the system. Simultaneous aliovalent substitution of Sr<sup>2+</sup> and Ti<sup>4+</sup> species by 10 % Pr<sup>3+</sup> and Al<sup>3+</sup> ions increases the temperature of a Pm3m – I4/mcm transition from 105 K in SrTiO<sub>3</sub> to ~250 K in Pr<sub>0.1</sub>Sr<sub>0.9</sub>Al<sub>0.1</sub>Ti<sub>0.9</sub>O<sub>3</sub>.

**Laser-neutron pump-probe experiment at the neutron time-of-flight spectrometer TOFTOF**

ID 175

**SCHWAIGER, Dominik (TUM Physik E13)***LOHSTROH, Wiebke (TUM - FRM2); MÜLLER-BUSCHBAUM, Peter (TUM Physik E13)*

Photosynthesis is one of the key mechanisms for “energy generation” in nature. It is controlled and driven by photoactive proteins. Their functionality is critically influenced by molecular movements in the range of ps – ns and sub-Å – nm. For comprehensive investigations of such systems it is necessary to gain information about the connection of their dynamics and functionality during and shortly after activation. The setup of the neutron time-of-flight spectrometer TOFTOF at MLZ-Garching is well suited for experiments, using quasielastic neutron scattering (QENS), in order to observe such, energetically low, motions in unordered materials. Therefore, at the instrument TOFTOF a novel sample environment system is to be installed. It will allow illumination of the sample during neutron measurement with light of specific wavelength. It is planned to apply the new system for photosensitive proteins, which undergo a photocycle, associated with the process of “energy generation”, triggered by the absorption of a photon. On the poster I will give an overview about the new setup and hopefully first results of measurement campaigns.



## Nanostructured TiO<sub>2</sub>/SnO<sub>2</sub> Templated by Amphiphilic Block Copolymer for Lithium-Ion Battery Anodes

ID 176

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Lithium-ion batteries (LIBs) have been widely used in many aspects of modern life. Compared with conventional graphite anodes, TiO<sub>2</sub> possesses superior cyclic stability and environmental benignity. However, its moderate conductivity and lithium ion migration ability still need to be improved to realize further development. As alternative anode material, SnO<sub>2</sub> afforded much higher theoretical specific capacity (1494 mAhg<sup>-1</sup>) as compared to TiO<sub>2</sub>. However, the big volume change and the formation of the thick SEI film during the cycling caused serious capacity recession. After comparing the characteristic of TiO<sub>2</sub> and SnO<sub>2</sub>, a novel TiO<sub>2</sub>/SnO<sub>2</sub> composite anode has been designed through amphiphilic block copolymer assisted sol-gel process in the present study. The morphology of the obtained TiO<sub>2</sub>/SnO<sub>2</sub> nanostructures are studied via scanning electron microscopy (SEM), the corresponding crystallization behavior is investigated with X-ray powder diffraction (XRD).

## Influence of benzocaine and propranolol on phospholipid bilayers

ID 179

**MANGIAPIA, Gaetano (German Engineering Materials Science Centre (GEMS) am Heinz Maier-Leibnitz Zentrum (MLZ))**

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Cell membranes play a fundamental role in protecting the cell from its surroundings, in addition to hosting many proteins with fundamental biological tasks. Drugs are able to perturb the structure of cell membranes, which can ultimately give rise to undesirable effects. Thus, a study of drug/lipid interactions is a necessary and important step in fully clarifying the role and action mechanism of active ingredients, and shedding light on possible complications caused by drug overdose. Here we present the results obtained in our research focused on the understanding of the influence of benzocaine and propranolol active principles on the structure of L- $\alpha$ -phosphatidylcholine-based membranes. The investigation has been performed by means of neutron reflectivity, grazing incidence small angle neutron scattering, and small/ultra-small angle neutron scattering. Investigations allowed discovering a stiffening of the membranes and the formation of stalks, caused by the presence of benzocaine. On the other hand, disordered bilayers (lamellar powders) and highly curved structures were found in the presence of propranolol. The results obtained may be rationalized in terms of the molecular structures of drugs and may serve as a starting point for explaining the toxic behavior in long-term and overdose scenarios.

## Formation and growth of mesoglobules in aqueous poly(N-isopropylacrylamide) solutions revealed with fast pressure jumps

ID 180

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Understanding of the kinetics of phase separation is of importance for numerous systems, for example to control demixing processes. The thermoresponsive polymer Poly(N-isopropylacrylamide) (PNIPAM) may serve as a model system to investigate the kinetics of phase separation. In aqueous solutions at temperatures above the cloud point, PNIPAM forms stable mesoglobules with a size and degree of hydration that depend strongly on pressure [1]. Previous time-resolved smallangle neutron scattering (TR-SANS) investigations of mesoglobule growth following a temperature change were hampered by the slow temperature equilibration [2]. To elucidate the formation and early stage growth of the mesoglobules in an aqueous PNIPAM solution, we applied TR-SANS after pressure jumps inducing the phase separation, enabling a time resolution of 50 ms. Three processes of mesoglobule growth are identified. During the first ~1 s, clusters of chains are formed which grow only slowly due to interconnecting chains. After disruption of these networks, mesoglobule growth is limited by diffusion and inner restructuring: Gradually, a dense polymer shell is formed that hinders the coalescence at later stages, resulting in a very slow growth.

[1] Niebuur, B.-J.; Papadakis, C.M. et al., ACS Macro Lett. 2017, 6, 1180

[2] Meier-Koll, A.; Papadakis, C.M.; Müller-Buschbaum, P. et al., Langmuir 2012, 28, 8791

## Crystal structure dependent solidification of undercooled glass-forming melts

ID 185

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Solidification from undercooled melts, either crystallisation or glass formation, is an important physical phenomenon of great technical and academic interests. Properties of the solid materials depend crucially on the solidification conditions. The competition between crystallisation and glass formation determines the stability of the undercooled melt and its glass forming ability. We investigate solidification behaviour of the congruent melting, glass-forming intermetallic melt Zr<sub>50</sub>Ni<sub>50</sub>. Employing electrostatic levitation (ESL) and high-speed video diagnostics, crystal growth velocity from the undercooled melt has been measured up to an undercooling of 300 K. Two distinct solidification behaviours of the Zr<sub>50</sub>Ni<sub>50</sub> melt have been identified, which differ in their recalescence temperature profile, growth velocity as a function of undercooling, and the resulting microstructure. With the help of in-situ synchrotron diffraction combined with ESL, we were able to associate the different solidification behaviours to two different phases nucleated from the undercooled melt: the stable orthorhombic ZrNi phase and the metastable cubic B2 phase. Both the stable and metastable phases occur statistically, independent of the degree of undercooling, and no difference in the liquid short range order can be detected intimately prior to solidification. We show that the slower growth of cubic phases might be a general feature in these Zr-based alloy melts, which could be one of the origins of their glass forming ability.

## Nuclear inelastic scattering for identification of iron ligand modes in dinitrosyl iron complexes and nitrogenase single crystals

ID 188

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Vibrational iron ligand modes of  $^{57}\text{Fe}$  labelled complexes can be accessed via synchrotron based nuclear inelastic scattering (NIS). First results of NIS experiments performed in October 2017 at PETRA III, DESY are presented and compared to simulations of the experimental partial density of vibrational states (pDOS). The influence of protonation on the vibrational properties of a dinitrosyl iron complex (DNIC) has been investigated. DNICs are a product of the reaction of the messenger molecule NO with iron sulfur clusters and their reactivity might depend on the protonation state. The density functional theory based simulations confirm the experimentally observed distinct differences between the two protonation states and reproduce the intense features in the pDOS energy region  $> 500 \text{ cm}^{-1}$  typical for Fe-NO vibrations [1]. Moreover orientation dependent NIS experiments on CO-free and CO-inhibited  $^{57}\text{Fe}$  enriched nitrogenase single crystals have been performed. Despite the low signal to noise ratio, slight but significant orientation dependent differences are observed for low energy modes  $< 100 \text{ cm}^{-1}$  as well as in the region of characteristic Fe-CO modes above  $460 \text{ cm}^{-1}$  [2].

[1] A. L. Speelman et. al., Inorg. Chem., 2016, 55, 5485.

[2] A. D. Scott et. al., J. Am. Chem. Soc., 2014, 136, 15942.

## Observing the structural evolution during chemical processes in-situ via SANS

ID 192

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The structures formed in self-assembling or self-organising systems can vary dramatically, and often non-linearly, depending on the processing conditions or as a result of external stimuli. In turn, these structural differences will often lead to pronounced differences in macroscopic properties and functionality. Optimisation of the functionality of such systems therefore entails increased control over the structure and consequently, a method of following the structural evolution during the synthesis or self-assembly process. To this end, we have developed a continuous-flow chemical reactor sample environment for SANS instruments, that enables uninterrupted observation of complex reactions with multiple reagents. Results from two preliminary experiments will be presented: one following the morphological evolution of micellar aggregates formed in an aqueous solution of a commercial surfactant with continuously varying pH and the other, a block copolymer synthesis exhibiting polymerisation-induced self-assembly. Both experiments show the structural changes in unprecedented detail and demonstrate the capability of following self-assembly processes and chemical reactions in situ on a SANS beamline. This is important as it expands the range of techniques available to probe complex soft-matter systems and opens the door to a variety of new measurements linking nano- and mesostructural phenomena to macroscopic properties.

## A split-and-delay unit for the European XFEL: Enabling hard x-ray pump/probe experiments at the HED instrument

ID 194

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For the High Energy Density (HED) instrument at the SASE2 - Undulator at the European XFEL an x-ray split-and-delay unit (SDU) is built covering photon energies from  $h\nu = 5$  keV up to  $h\nu = 24$  keV. This SDU will enable time-resolved x-ray pump / x-ray probe experiments as well as sequential diffractive imaging on a femtosecond to picosecond time scale. Further, direct measurements of the temporal coherence properties will be possible by making use of a linear autocorrelation. The x-ray FEL pulses are split by a sharp edge of a silicon mirror (BS) coated with Mo/B<sub>4</sub>C and W/B<sub>4</sub>C multilayers. Both partial beams then pass variable delay lines. For different wavelengths the angle of incidence onto the multilayer mirrors will be adjusted in order to match the Bragg condition. Because of the different incidence angles, the path lengths of the beams will differ as a function of wavelength. Hence, maximum delays between 1.0 ps at  $h\nu = 24$  keV and up to 23 ps at  $h\nu = 5$  keV are possible.



## Scanning X-ray Diffraction Microscopy of Ion Irradiated VO<sub>2</sub>

ID 195

**JOHANNES, Andreas (ESRF)**

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Vanadium dioxide undergoes a phase transition from an insulating, monoclinic (M1) phase at low temperatures to a metallic, rutile (R) phase above 68 °C. Since the two phases show distinctly different electrical conductivity and absorption in the near infrared VO<sub>2</sub> is interesting for “smart” electrical and optical components or window coatings. Ion beam irradiation decreases the transition temperature, making it an interesting tool for technological application, esp. for controlled patterning when considering the high special resolution and flexibility obtainable with focused ion beam (FIB) systems. Here we present scanning X-ray diffraction microscopy results on two distinct sample arrangements of VO<sub>2</sub>. First, single crystalline VO<sub>2</sub> microwires were investigated. They clearly show patterned FIB irradiation induced strain patterns and the coexistence of metallic and insulating phases in very close proximity within a single crystal. Secondly, a nanocrystalline thin film of VO<sub>2</sub> was nanostructured with FIB irradiation. We show that for a given film thickness an optimal irradiation dose yields a high contrast in the phase transition. In this patterned thin film it seems that the boundary lines between patterned surfaces preferentially follow the granularity of the film.



## Operando X-ray absorption spectroscopy (XAS) study of the selective CO methanation on Ru/TiO<sub>2</sub> catalysts: TiO<sub>2</sub> surface morphology effects

ID 197

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Metal-support interactions in Ru/TiO<sub>2</sub> catalysts have a decisive impact on their catalytic activity and selectivity for CO methanation. These effects and the catalytic performance of the catalysts were found to change upon varying the surface area of the support, which is not yet well understood. One approach to explain this change would be a change in the surface morphology of the TiO<sub>2</sub> crystallites. In this study, we investigated Ru/TiO<sub>2</sub> catalysts with different TiO<sub>2</sub> morphologies for the selective CO methanation, employing anatase TiO<sub>2</sub> nanocrystals with preferentially {001}, {100}, and {101} oriented facets. The results indicate a significant impact of the TiO<sub>2</sub> surface morphology on the initial activation phase of the catalyst and its long-term stability. The Ru/TiO<sub>2</sub>-{001} catalyst reached its highest activity already in 5 min, then it started to continuously deactivate. For Ru/TiO<sub>2</sub>-{101} and Ru/TiO<sub>2</sub>-{100}, the activation was much slower, taking 150 and 300 min, respectively. Furthermore, these catalysts were very stable during >1000 min on stream. Based on operando XANES measurements, it could be demonstrated that under reaction condition ~ 95% of the Ru NPs on TiO<sub>2</sub>-{001} become metallic in 10 min, while it takes up to 80 min to reach the same state for TiO<sub>2</sub>-{100}. These observations indicate strong effects of the TiO<sub>2</sub> surface morphology on the electronic properties of the supported Ru NPs.

## Morphology of printed active layers for organic solar cells as studied with advanced scattering techniques

ID 198

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*MÜLLER-BUSCHBAUM, Peter (TU München)*

Bulk heterojunction (BHJ) organic solar cells have gained significant improvements via novel organic synthesis methods and optimized fabrication routes, especially with respect to their potential roll-to-roll processing for large-area device manufacturing. Printing techniques allow for upscaling to industrial-oriented scale which is not the case for laboratory deposition techniques like spin coating. Thus, roll-to-roll processing on flexible substrates is very attractive to be implemented as production techniques for organic solar cells. Several methods of printing techniques can be used to reach this goal, such as for example slot-die coating. In a BHJ polymer structure, the morphology plays an important role for the device efficiency. Different factors can influence the morphology of the active layer. For example, the different ratio of donor material and acceptor material can influence the nanostructure and the phase separation of the active layer. In our experiment, the active layers were fabricated with different ratios of low-bandgap polymer PTB7 and fullerene derivate PCBM using the slot-die printing technique. The morphology and structure of printed active layers are investigated by atomic force microscopy (AFM), scanning electron microscopy (SEM) and grazing incidence small angle x-ray scattering (GISAXS).

## The low flux neutron source AKR-2

ID 202

*BERNT, Nico (TU Dresden – Training Reactor AKR-2)*

*LANGE, Carsten (TU Dresden – Training Reaktor AKR-2)*

The training and research reactor AKR-2 is a thermal, homogeneous, solid material moderated zero power reactor with maximum permanent power of 2 Watt. AKR-2 was completely refurbished in 2005 and is actually 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://tudresden.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 in radiation protection. 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. In the presentation, we will give a detailed description of the AKR-2's design and its physical properties. Some selected results of neutron imaging with thermal neutrons which was performed for the first time at AKR-2 will be shown. Furthermore, we would like to discuss the question how the AKR-2 can contribute in the education of scientists dealing with neutron scattering methods in future?

**Characterization of organic thin films for potential application in energy conversion devices**

ID 203

**WEINDL, Christian (TUM Physik)***LÖHRER, Franziska (TUM Physik); MÜLLER-BUSCHBAUM, Peter (TUM Physik)*

For years, carbon nanostructures have played a prominent role in case of electrochemical energy storage. Especially carbon nano onion films as a conductive additive for supercapacitors can be implemented to improve electrical characteristics. These onions with an almost spherical shape consist of several enclosed fullerene-like carbon shells. Amongst various ways to synthesize these onions, the most commonly used technique is the graphitization of nano diamonds at temperatures above 1000°C. [1] These high temperatures lead to a high degree of sp<sup>2</sup>-hybridization and thereby, result in comparatively high electrical conductivity values, a large surface area and nanoscopic size. [2] However, not much is yet known about the synthesis and morphology of thin films with implemented carbon onions. Therefore, we aim to investigate the influence of different processing pathways on the structural and optoelectronic properties of these films. In first studies, we optimize processing parameters such as the type of solvent, concentration and spin coating and probe the resulting morphology using surface imaging as well as synchrotron-based X-ray scattering techniques.

[1] M. Zeiger, N. Jäckel, V. N. Mochalin, V. Presser, J. Mater. Chem. A, 2016, 4, 3172-3196

[2] M. Zeiger, N. Jäckel, D. Weingarh, V. Presser, Carbon, 2015, 94, 507-517

## Supramolecular structure of monohydroxy alcohols

ID 205

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Monohydroxy alcohols have been scrutinized as a model of hydrogen bonded fluids. These hydrogen bonds are essential for the structure and dynamics of water, aqueous solutions and alcohols [1]. Monohydroxy alcohols are supposed to form supramolecular structures via hydrogen bonding in the liquid phase. We have investigated various alcohols by X-ray diffraction (XRD) over a temperature range from 175K to 405K at beamline BL9 at synchrotron light source DELTA (TU Dortmund). The supramolecular structures of 2E1H (2-ethyl-1-hexanol) and 4M3H (4-methyl-3-heptanol) mixtures and pure monohydroxy alcohols such as 6M3H (6-methyl-3-heptanol) and 5M3H (5-methyl-3-heptanol) were studied. Dielectric spectroscopy studies suggest ringlike arrangements for 4M3H and 5M3H (200K-300K), while chain arrangements were observed at 2E1H and 6M3H (175K-275K) [2]. We will discuss the results of the XRD measurements in relation to the supramolecular structures and compare them with the results of dielectric spectroscopy.

[1] Kaatze, U., et al. "Hydrogen network fluctuations and dielectric spectrometry of liquids". *Journal of Non-Crystalline Solids*. (2002), 305(1), 19-28.

[2] Singh, L. P., et al. "Dynamics of glass-forming liquids. XVII. Dielectric relaxation and intermolecular association in a series of isomeric octyl alcohols." *The Journal of chemical physics*. (2013), 139(14), 144503.



## SAPHiR, the instrument for neutron science at high P and T

ID 209

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*KEPPLER, Hans (BGI, Uni Bayreuth)*

SAPHiR, the Six Anvil Press for High Pressure Radiography and Diffraction is a new instrument at the FRM II dedicated to neutron science at extreme pressure and temperature conditions. The three-axis multianvil press has a combined pressing force of 2400 tons (24 MN) and can perform experiments at up to 15 GPa in samples with volumes of 10-30 mm<sup>3</sup>. Applications include phase transformations at high P and T, reaction kinetics, crystallography of hydrogen bearing high pressure phases, high resolution radiography, and rheological measurements for solid state physics, chemistry, material and geoscience. SAPHiR will use a thermal neutron beam that is focussed on the sample position by an elliptic neutron guide. The detector system consists of wave-lengthshifting- fibre detectors in the backscatter regime and helium-3 detector banks at 90° from the incident beam and in the forward scatter regime. The WLSF detectors combine a superior position resolution with a good detector efficiency > 50 %, which allows measurements with a  $\Delta d/d$  resolution in the 10<sup>-3</sup> range. The <sup>3</sup>He detectors are composed of 660 individual PSD tubes that combine a high detection efficiency with a low gamma sensitivity. In combination the detector system can measure d-values of ~0.6 – 15 Å. User access will commence once the infrastructure in the eastern neutron guide hall is completed.

## GISAXS reconstruction of grating profiles produced using self-aligned multiple patterning

ID 213

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*SOLTWISCH, Victor (Physikalisch-Technische Bundesanstalt (PTB)); KLINE, R. Joseph (National Institute of Standards and Technology (NIST)); KRUMREY, Michael (Physikalisch-Technische Bundesanstalt (PTB))*

New approaches are needed for the fast, non-destructive dimensional measurement of complex nanostructures in the semiconductor industry. One technique being considered is Small-Angle X-ray Scattering (SAXS), which has already been used in transmission geometry to reconstruct the line profile of gratings with low uncertainties. Grazing-Incidence SAXS (GISAXS) in reflection geometry additionally provides surface sensitivity, but the interpretation of the scattering is complicated by multiple scattering effects. In order to produce structures beyond the diffraction limit of a single lithographic image, selfaligned multiple patterning can be used. In self-aligned multiple patterning, sidewalls are deposited on the original line and the original line is removed, such that the sidewalls form lines with a doubled structure density. If the sidewall width and the original linewidth do not match, an alternating pitch error is introduced, impacting the performance of the resulting structures. We present GISAXS measurements of a series of grating samples with a nominal pitch of 32 nm. The samples were produced by self-aligned multiple patterning, and different pitch errors were introduced on purpose. From the intensities of the grating diffraction orders we quantify the pitch errors and compare our results to previous transmission SAXS measurements of the same samples.

## Effect of inorganic SnIP nanoparticles on the morphology of polymer blends for photovoltaic applications

ID 215

**GROTT, Sebastian (TUM)**

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Organic solar cells have attracted increased attention due to their advantages in tunable characteristics, low-cost manufacturing processes and flexibility, which opens up a promising alternative for conventional photovoltaics. Recently, the most widely investigated bulk heterojunction donoracceptor system of P3HT:PCBM was doped with iron oxide nanoparticles, resulting in an increased efficiency.[1] Based on this approach, we investigate the effect of doping P3HT:PCBM active layers with inorganic SnIP nanoparticles[2]. We study the influence of different inorganic SnIP nanoparticle concentrations on the inner morphology of the polymer thin films by using grazing incidence wide angle X-ray scattering (GIWAXS). These results will be compared to the photoelectric characteristics of the corresponding organic solar cells.

[1] D. M. González, et al., Adv. Energy Mater. 2015, 5, 1401770.

[2] D. Pfister, et al., Adv. Mater. 2016, 28, 9783.

## Spin dynamics and anomalous anisotropy gap in the metallic perovskite SrRuO<sub>3</sub>

ID 220

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SrRuO<sub>3</sub> is one of the very few perovskite metallic ferromagnets; it exhibits anomalous transport, an invar effect, non-Fermi liquid behavior, a magnetic shape-memory effect and it is an important substrate for various oxide heterostructures. Strong spin-orbit coupling (SOC) is visible in the invar effect and the large magnetic anisotropy. Recently, we could grow large single crystals of SrRuO<sub>3</sub> using the floating-zone technique in an image furnace [1,2]. We report the first inelastic neutron scattering study of the spin dynamics on single crystals. By detwinning the strongly twinned crystals with a magnetic field, it was possible to investigate the dispersion in two orthorhombic directions. Our results yield the expected quadratic spin wave dispersion of a ferromagnet. However the stiffness constant considerably deviates from an earlier inelastic neutron scattering study on powders and it increases with increasing temperature towards TC. We also find a non-monotonous temperature dependence of the anisotropy gap which again disagrees with the powder results. The possible relation of the gap and stiffness parameters of the magnon dispersion with Weyl modes will be discussed.

[1] S. Kunkemöller et al., Chrys. Res Tec. 51, 299 (2016)

[2] S. Kunkemöller et al., PRB 96, 220406(R) (2017)

[3] S. Itoh et al., Nat. Commun. 7, 11788 (2016)

## Morphology Control of Low Temperature Fabricated ZnO Nanostructures for Transparent Active Layers in All Solid-State Dye-Sensitized Solar Cells

ID 225

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Dye-sensitized solar cells (DSSCs) offer outstanding potential due to their minor investment compared to inorganic solar cells and higher stability compared to organic solar cells. In this kind of cells, nanostructured inorganic metal oxides with tunable morphologies are of great importance. Among the many inorganic metal oxides, ZnO has been widely explored due to its outstanding electrical and optical properties and rich variety of morphologies. In order to improve the interfaces between ZnO and p-type polymers, routes to tune the length scales of the nanostructures are explored. The morphologies are probed using scanning electron microscopy (SEM) and grazing incidence small-angle X-ray scattering (GISAXS). Through GISAXS measurement, the inner morphology, which is crucial for application in DSSCs, is probed. Based on the controlled nanostructured ZnO films, solid-state dye-sensitized solar cells (ssDSSCs) are prepared, for which every layer is deposited at low temperature to reduce the energy consumption of the manufacturing process. Transparent active layers for ssDSSCs are obtained, which demonstrates the possibility for building integrated solar cells.

## Precision determination of the axial-vector coupling constant from neutron beta decay

ID 226

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Within the standard model of particle physics only two free parameters determine the decay of the free neutron, where we profit from the precise determination of the Fermi coupling constant in muon decay. These free parameters are the ratio of axial-vector and vector coupling constants  $= g_A/g_V$  and the CKM mixing matrix element  $V_{ud}$ . With about a dozen experimental observables the problem of the determination of these parameters is largely over-constrained and hence enables the search for new physics via non  $V-A$  couplings in this process. We present the result of the neutron decay spectrometer Perkeo III on the ratio of coupling constants which was derived from a measurement of the parity violating beta asymmetry in polarized neutron beta decay with a precision of  $\Delta = 5.5 \times 10^{-4}$ . This result is more precise than the current PDG world average by a factor of four and clarifies a long standing tension between previous measurements. The instrument was installed and operated at the Institut Laue-Langevin. A pulsed cold neutron beam was used to control or eliminate major sources of systematic error. We will discuss the measurement and first implications of the result on the search for new physics. The presentation will conclude with a status report on the follow-up instrument PERC, which is currently under construction at the beamline MEPHISTO of the MLZ. The aim of PERC is an improved measurement of several decay correlations in neutron beta decay by an order of magnitude.



## Role of native oxide layer in silicon anodes for Li/S batteries

ID 227

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Lithium/sulfur (Li/S) and Lithium/air systems are possible candidates for future electrochemical energy storage, due to higher gravimetric density compared to conventional Li-ion batteries. In these systems, a lithiated silicon (Si) electrode often replaces the Li metal anode, used as a Li ions reservoir. Detailed studies on lithiation and delithiation of Si anodes are of fundamental importance in understanding capacity fading effects. Silicon crystals are used with the native silicon dioxide (SiO<sub>2</sub>) layer: upon lithiation and delithiation, Li ions interact first with the SiO<sub>2</sub>, and subsequently intercalate in the Si crystal forming a Li/Si alloy. Due to the negative scattering length density of Li, neutron reflectometry is a powerful technique for investigating lithiation and delithiation of Si anodes. Two different Li/Si cells were prepared, one with and one without native oxide layer (removed using HF etching) on the Si anode. Both were measured at the reflectometer V6 at the BER II neutron source, to shed some light on the role of the SiO<sub>2</sub> native layer formation and its (ir-)reversibility using both electrochemical (CV, EIS, charge/discharge) and in situ/operando neutron reflectometry measurements.



## Colloidal quantum dots solids for photovoltaics

ID 229

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Colloidal quantum dots (CQDs) have been attracted many attentions on various electronic applications, like light emitting diodes (LEDs), photo detectors (PDs), photovoltaics (PVs) etc. due to their unique intrinsic properties, like tunable energy band-gap, stability against ambient circumstance. Comparing with cadmium chalcogenides CQDs, the lead chalcogenides CQDs reveal much weaker exciton binding energy because of the smaller effective electron mass, which is beneficial for the extraction of electrons and therefore suitable for applications in PVs rather than LEDs. The functional CQDs' array films made by one of various solution processes, like spin coating, spray-coating or dip coating in room conditions would inevitably have disordering film structure and bring in many trap states. The thermal treatment is a regular method in organic PV device fabrication for the annealed crystalline of polymer but rarely used in CQDs film for fabricating related devices. In this work, we proceeded the thermal treatments on our CQDs' films with different temperatures and used grazing incidence small angle / wide angle x-ray scattering (GISAXS/ GIWAXS) to investigate the inner structure of thermal treated CQDs' films, including the superlattice structure, inter dot spacing and the Nano-crystals orientations. We also used pump-probe transient absorption (TA) spectroscopy to observe the order of energy distribution and qualitatively evaluate the yield of generated excitons.

## Specialized Sample Environment at BESSY II

ID 234

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The sample environment activities play an increasing role in the BESSY II user service. The sample environment group, originally based at the BER II neutron source, is now fully established at BESSY II. It provides and supports various sample environment equipment for experiments at the source. In addition, it supports beamline scientists and synchrotron users in the design, construction, commissioning, and improvement of sample environments, especially in the field temperature, pressure, and electro-magnetic-field regulation methods. We present some highlights of recent sample environment developments at HZB, i.e. for in-situ gas adsorption studies combined with SAXS, XRD and EXAFS, for future XES investigations at cryogenic temperatures, as well as dedicated sample holders for improved macromolecular crystallography, and specialized fluid cells for UHV-beamlines.

## X-ray magnetic linear dichroism as a probe for non-collinear magnetic states in a DyCo<sub>5</sub> single ferrimagnetic layer

ID 236

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We report on exploiting the X-ray magnetic linear dichroism (XMLD) contrast for probing the noncollinear states in a DyCo<sub>5</sub> ferrimagnetic thin film. Utilizing x-ray magnetic circular dichroism which is sensitive to the magnetization of one elemental sublattice, an anomalous 'wing shape' hysteresis loop is observed slightly above its compensation temperature. Based on the hysteresis shape, it is inferred the occurrence of an out-of-plane partial domain wall formation between surface and towards the bulk. By taking advantage of the strong linear dichroism of the Dy element at the M<sub>5</sub> absorption edge, the formation of this partial domain wall is directly observed via XMLD contrast as a function of the magnetic field. This demonstrates the occurrence of a non-collinear magnetic transition at high magnetic fields of about 6T for this system. The measurements were performed with the VEKMAG end station at BESSY II (Helmholtz-Zentrum Berlin).

## Combining x-ray emission and x-ray Raman spectroscopy for the study of Earth materials at high pressure and high temperature

ID 245

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X-ray emission and x-ray Raman scattering spectroscopy are powerful tools to investigate the local electronic and atomic structure of high and low Z elements in situ and can be applied at high pressure and high temperature conditions which are present in e.g. the Earth's mantle. We developed a setup for simultaneous x-ray emission (Fe K $\beta$ <sub>1;3</sub> and valence to core) and x-ray Raman scattering (low Z elements' absorption edges) studies at beamline P01 of PETRA III synchrotron radiation source using a wavelength dispersive von Hamos spectrometer together with the existing multiple-analyzer Johann-type spectrometer in combination with a laser heating device. It's capabilities are demonstrated by investigating the iron spin crossover in siderite (FeCO<sub>3</sub>) and bridgmanite ((Mg,Fe)SiO<sub>3</sub>). This setup provides a unique combination in order to achieve new insights into the spin transition and compression mechanisms of mantle materials which is of importance for the understanding of the macroscopic physical and chemical properties of the inner Earth.



## In situ neutron diffraction study of lithium-ion batteries during operation and relaxation

ID 247

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Reduced capacity and rate capability, lithium plating and current collector corrosion are some of the effects detrimental to lithium-ion battery performance and safety at low temperature operation. Novel electrode designs and electrolytes that suit these conditions are currently investigated. Detailed knowledge of the effect of low temperature conditions on the battery during operation is helpful for further improvements. In operando visualization of the impact on electrode inhomogeneity can illustrate shortcomings in current electrode design. We performed time resolved in situ neutron diffraction measurements on custom made NCM/graphite pouch cells at STRESS-SPEC, MLZ. The batteries showed a lithiation gradient in the graphite anode during operation and subsequent relaxation. Diffraction data of the discharge and relaxation processes is presented and the effect of active material loading and electrode morphology on anode inhomogeneity are discussed.

## Complementarity of Neutrons and X-rays on the Example of Hydrocarbon/Fluorocarbon Small Unilamellar Vesicles

ID 248

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The self-assembly of small unilamellar vesicles (SUVs) in mixtures of perfluorinated and hydrocarbon surfactants has been subject of intense studies in the past with a focus on the phase diagram [1,2], shape and kinetics of the aggregates depending on concentration and mixing ratio [3] as well as control thereof [4]. Given that the scattering length densities (SLD) of perfluorinated and hydrocarbon surfactant tails differ largely for both neutron and x-ray scattering, SANS and SWAXS are very sensitive to the composition as well as the internal structure of the bilayer [5]. Using contrast-variation SANS (KWS-1, FRM II), we were recently able to show that the composition of our SUVs differs significantly from the sample composition even for a nearly symmetric mixture (mole fraction xTDMAO = 0:43). We are currently improving our structural model based on this finding in order to fully exploit the detailed structural information contained in our stopped-flow SAXS/WAXS data (ID02, ESRF) [6]. These results then are correlated with a thermodynamic analysis of the system.

[1] C. Wolf et al., *Langmuir* 25(19):11358, 2009.

[2] K. Bressel et al., *Soft Matter* 7(23):11232, 2011.

[3] J. Gummel et al., *Soft Matter* 7(12):5731, 2011.

[4] K. Bressel et al., *ACS Nano* 6(7):5858, 2012.

[5] T. Narayanan et al., chap. 7 in *Advances in Planar Lipid Bilayers and Liposomes*, vol. 20, Academic Press 2014.

[6] A. F. Hörmann et al., in preparation.



## Resonant X-ray Diffraction at the Petra III Chemical Crystallography Beamline

ID 251

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Since 2010, the joint research project “Chemical Crystallography Beamline” is working together to build up a dedicated experimental station at DESY’s PETRA III east extension in Hamburg. The partners from Freiberg contribute in particular by establishing the setup for Resonant X-Ray Diffraction methods. In addition to energy resolved photon detection and polarization analysis, this setup comprises the application of physical fields as well as temperature, which specifically covers the design and construction of a customized sample chamber for in-situ characterization of pyro- and piezoelectrical properties. This chamber allows, for instance, the combination of pyroelectric measurements with the newly developed Resonant X-ray Diffraction Method RSD (Resonantly Suppressed Diffraction), which can evaluate atomic displacements in the pico-meter range. Further subjects within the sub-project are the characterization of dynamical processes and phase transformations with a focus on materials for energy conversion and storage. Respective results of recent research include on the one hand pyroelectricity in polar phases of strontium titanate  $\text{SrTiO}_3$  and the standard pyroelectric structures lithium niobate  $\text{LiNbO}_3$  and lithium tantalate  $\text{LiTaO}_3$ , and on the other hand atomic displacements in yttrium-manganese-oxide  $\text{YMn}_2\text{O}_5$  and rare-earth oxoborates  $\text{RX}_2\text{Z}_2\text{O}(\text{BO}_3)_3$  (R = rare earth, and X, Z = Ca).



## Investigation of organically linked iron oxide nanoparticle supercrystals using SAXS and SANS

ID 254

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The particular innovative potential of the SFB 986 is its ability to develop macroscopic materials – structured in a multi-scale way, designed “on the drawing board”. Scattering techniques are a powerful tool to investigate these materials on several length scales. Due to their different sensitivity to the included phases, SAXS (P07 at PETRA III, Hamburg) and SANS (SANS-1 at FRM II, Garching) were used to characterize the structure of organically linked iron oxide nanoparticle supercrystals. In addition, the self-assembling process of the coated nanoparticles and the materials behaviour during a heat treatment were studied in situ. The results of these experiments were used to optimize the production process of the hierarchical materials system.

## Temperature dependent study of the local structure of bromine ions in polymerized ionic liquids

ID 255

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Understanding the structural foundation governing the charge transport is the key for any rational design of electrochemical materials. In this respect, amorphous conductors such as ionic liquids (ILs) and polymer electrolytes are well investigated. At variance with other materials, the polymerized ILs (PolyILs) are prepared through direct covalent bonding of functional monomers containing IL fragments, hence combining the benefits of ILs in terms of high charge density with those of polymers in terms of mechanical stability. In a recent work the ionic diffusivity of several PolyILs was investigated by means of dielectric spectroscopy combined with nuclear magnetic resonance and differential scanning calorimetry (C. Gainaru et al., J. Phys. Chem. B 120, 2016). Based on the results, we propose a new approach to estimate single-ion diffusivity from the conductivity relaxation by connecting the elementary diffusion step of the ions with structural details. In order to probe our estimates, we performed EXAFS experiments at beamline BL10 of DELTA and P64 of PETRA III at the Br- K-edge on monomer and polymer ILs consisting of an imidazolium ring as cation and bromine as anion to reveal temperature-induced changes in bromine's local coordination and connect it to the ionic diffusivity. The measured spectra exhibit major structural differences between the two types of ILs, an exciting result which opens new venues for the understanding of charge transport in concentrated electrolytes.

## Inelastic neutron scattering studies of magnons in the conical and field-polarized phase of MnSi

ID 258

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Various chiral spin structures, stabilized by the antisymmetric Dzyaloshinsky-Moriya interaction (DMI), are known to occur in the cubic chiral magnet MnSi. Strongly coupled, low-energy helimagnon bands are a universal characteristic of chiral systems and were studied in the presence of multiple domains [1] and the single domain phase [2]. Furthermore, a non-reciprocal magnon dispersion  $E(\mathbf{q}) \neq E(-\mathbf{q})$  was observed in MnSi [3]. We present a comprehensive study of the magnetic field dependence of the magnon dispersion covering the helical phase and the field-polarized phase. The related inelastic neutron scattering experiments have been conducted at the cold triple-axis spectrometer MIRA (FRM2) and TASP (PSI). Our study determines the evolution of the magnetic structure factor across the continuous phase transition at the critical magnetic field  $H_{c2}$ , notably its non-reciprocity [4]. Using the theoretical framework [5], developed by M. Garst, we calculated the dispersion and spectral weights of the probed magnon branches. Factoring in the instrument resolution, we achieved excellent agreement between the theoretical predictions and the experimental results [4, 6].

[1] M. Janoschek et al. Phys. Rev. B, 81:214436, Jun 2010

[2] M. Kugler et al. Phys. Rev. Lett., 115:097203, Aug 2015

[3] T. J. Sato et al. Review B, 94(14):144420, Oct 2016

[4] T. Weber et al. submitted, arXiv:1708.02098

[5] M. Garst et al. Journal of Physics D, 2017

[6] T. Weber et al. SoftwareX, 2016

## Ultra small iron nanoparticle superlattice on graphene on iridium

ID 259

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The physical properties of ultra small clusters, such as magnetism, can differ fundamentally from the bulk material and are dominated by their confinement and atomic structure [1, 2]. The arrangement of such nanoparticles in a superlattice not only allows to reach high density and small size distribution, but it also enables new characterization approaches [2]. This is of great interest for magnetic storage applications, as well as fundamental science. Iridium seeding on graphene on Ir(111) allows to grow large area ultra small ~1nm iron nanoparticle superlattices [1]. We studied the system with various measurement techniques in-situ on different setups in order to gain and correlate structural, electronic and magnetic information. Structural characterization of the lattice from scanning probe techniques can therefore be combined with atomically resolved surface x-ray diffraction [2]. We have observed a size dependent structural phase transition which we will link to magnetic information from x-ray magnetic circular dichroism. An outlook on tailored substrates [3] and the possibility of coverlayers will be given.

[1] Alpha T. N'Diaye, et al, New Journal of Physics 11, 103045 (2009).

[2] Dirk Franz, et al, Phys. Rev. Lett. 110, 065503 (2013).

[3] Arti Dangwal Pandey, et al, J. Appl. Phys. 120, 075304 (2016).

## Hard x-ray photoemission spectroscopy of in operando strained Vanadiumdioxide films on PMN-PT

ID 260

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VO<sub>2</sub> films on the relaxor ferroelectric Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)<sub>0.72</sub>Ti<sub>0.28</sub>O<sub>3</sub> (PMN-PT) provide a promising candidate for the realization of a “Mott-tronic” device. VO<sub>2</sub> undergoes a first-order structural phase transition at about 340 K and simultaneously switches from insulating to metallic behavior by a five orders of magnitude resistance drop. Importantly, the insulator-to-metal transition can also be driven by out-of-plane compressive lattice strain as being mediated, for example, by a PMN-PT substrate. Here, we present a hard x-ray photoelectron spectroscopy (HAXPES) study of the electronic structure of VO<sub>2</sub>/PMN-PT interfaces across the strain- and temperature-induced phase transition. The in operando monitoring of the shapes and positions of characteristic core-level emissions directly reveals strain-dependent changes of the electronic structure and phase transition temperature of the VO<sub>2</sub> film as well as bias-dependent changes of the electronic energy-level alignment at the VO<sub>2</sub>/PMN-PT interface. Overall, our results establish HAXPES as a powerful tool for the in operando investigation of functional oxide interfaces.



## Exchange coupling effects in hybrid Gr-4f RE systems

ID 261

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Hybrid Ferromagnetic/Graphene (FM/Gr) systems enclose remarkable technological opportunities by bridging spintronics with promised ultra-fast Gr-based electronics and photonics. These are also of fundamental relevance since Gr actively interacts with the neighboring materials determining a modification of the electronic and magnetic properties of the system. In particular, Graphenespaced magnetic systems with antiferromagnets offer exciting opportunities for the investigation of exchange-coupling phenomena in spintronics. We have recently shown that ultra-thin graphene/Co films grown on Ir(111) or Pt(111) templates exhibit robust perpendicular magnetic anisotropy (PMA) and antiferromagnetic exchange-coupling when Fe is deposited on top of Gr. These results gather a collection of magnetic properties well-suited for applications. However, one drawback in 3d-FM/Gr systems, is that the FM-Gr hybridization is so strong that it impacts Graphene's electronic properties, for example, the presence of Dirac's cone. Instead, the weak interaction between 4f-FM and Gr preserves Gr's unique electronic structure and particularly Dirac's cone. Here, by resorting to X-ray absorption and magnetic dichroism (XAS-XMCD) measurements we investigate the magnetic configurations, the nature of the Gr-mediated exchange coupling and the magnetic anisotropy in 4f-FM/Gr hybrid systems such as Eu, Dy or Ho as an extension of Gr-synthetic antiferromagnetic (SAF) systems.



## Exhaustive X-ray Crystallographic Screening of a Hit-Enriched 96 Fragment Library Against Diverse Targets

ID 262

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Modern automated beamlines are well suited for crystallographic screening of 100-500 entry fragment libraries or diverse subsets at no higher effort than alternative biochemical or biophysical pre-screening assays. As an entry point for a direct crystallographic fragment screening and drug discovery, we compiled 96 well-suited fragments based on experience from prior fragment screening campaigns and PDB entries. For evaluation, we crystallographically screened this library against seven diverse targets (endothiapepsin, protein kinase A, tRNA-guanine transglycosylase, carbonic anhydrase II, thrombin, 17- $\beta$ -hydroxysteroid-dehydrogenase 14 and thermolysin) at different conditions. Crystallographic hits were obtained for each target at hit rates up to 31%. We compare the fragment-bound structures of each target. Fragments bound to multiple targets are compared with respect to their binding mode and local interaction pattern. We also show the potential to follow up on these fragments based on feasible growing vectors in the fragment-bound structures. The presented library is available in collaboration with Jena Bioscience as a ready-to-soak 96-well plate (Frag Xtal Screen). Complementary fragment libraries and methods are part of the Frag2Xtal and Frag4Lead service facility for crystallographic fragment screening currently made available and extended at the automated crystallographic BL14.2 at the BESSY II storage ring of the Helmholtz-Zentrum Berlin.

## Investigation of metastable precipitates in Ti-15Mo by in-situ SANS

ID 266

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Titanium alloys have plenty of applications in industry and medicine due to unique combination of high strength, low density, and excellent biocompatibility [1]. Here, we would like to demonstrate results of investigations of Ti-15Mo (wt.%) alloys using small-angle neutron scattering (SANS). These alloys contain metastable  $\omega$  (hexagonal) and  $\alpha$  (hcp) precipitates in  $\beta$ -phase matrix. The resulted microstructure has grate impact on mechanical properties and thermostability of the material. SANS data were acquired at three directions of the single crystal sample – [111], [110] and [100] of  $\beta$ -phase. Heating rates of 1K/min and 5 K/min were applied for the in-situ measurements. Observed spots at 2D SANS patterns at temperatures lower than 560 0C were formed by isothermal  $\omega$  precipitates arranged in simple cubic structure. Increasing of temperature leads to growing of volume fraction of these  $\omega$  precipitates and increasing of mean interparticle distance.  $\Omega$  phase became invisible at maximum instrumental resolution then temperature approach 580 0C and simultaneously slightly stronger scattering was detected from very long  $\alpha$  particles. Structure of these  $\alpha$  precipitates was not changed during cooling down from 600 0C to room temperature

[1] Lutjering G, Williams JC (2007) Titanium. Engineering materials, processes. Springer, Berlin, pp 1–39. doi:10.1007/978-3-540-73036-1.

## Backfilling of Mesoporous Titania Structures with Heavy Element Containing Small Molecules and High-Efficiency Polymer PTB7-th

ID 268

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The future-shaping properties of next-generation solar cells can potentially be achieved through an inorganic-organic hybrid photovoltaics (HPV) approach. Essential for a functioning HPV device is efficient charge separation at the interface between inorganic and organic material. Due to titania being a wide-bandgap semiconductor and having good electron mobility, it is viable as a mesoporous inorganic structure to be infiltrated by an organic hole-conducting material. On the organic part, the high-efficiency, hole-conducting polymer PTB7-th is infiltrated into titania films to form the active layer of the photovoltaic device. Additionally, a novel, heavy element containing polymer molecule (Phen-Te-BPinPh) has been synthesized and properties with regard to photovoltaic applications are characterized. To further enhance the solar cell performance, Phen-Te-BPinPh could be implemented either by backfilling into a layered setup between the PTB7-th and the mesoporous TiO<sub>2</sub> layer or as a dopant into the PTB7-th. In determining the backfilling efficiency of PTB7-th and Phen-Te-BPinPh into the mesoporous titania matrix, Time-of-Flight Grazing Incidence Small Angle Neutron Scattering (ToF-GISANS) is the most valuable and significant method of characterization. [1]

[1] M. Rawolle, K. Sarkar, M. Niedermeier, M. Schindler, P. Lellig, J. Gutmann, J-F. Moulin, M. Haese-Seiller, A. Wochnik, C. Scheu, and P. Müller-Buschbaum, *ACS Applied Materials and Interfaces*, 2013, 5(3), 719-729.



## Formation of a micrometer positron beam at the Scanning Positron Microscope

ID 273

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Positron annihilation lifetime spectroscopy (PALS) is a powerful tool in a wide range of material science. To investigate inhomogeneous defect distributions, e.g. close to fatigue cracks or dispersive alloy, with PALS a monochromatic pulsed positron beam of variable energy with a diameter in the range of 1  $\mu\text{m}$  and a pulse width of 150 ps FWHM is needed. In order to achieve this beam quality the Scanning Positron Microscope (SPM) was developed and built at the Universität der Bundeswehr München. The limit of low count-rates and corresponding exceedingly long measurement times is overcome transferring the SPM to the intense positron source NEPOMUC at the MLZ in Garching. To connect the SPM to NEPOMUC a special interface has been constructed. A new developed positron radio frequency elevator has been tested, which is the final step of the SPM interface. The elevator compensates the loss of potential energy, lost by the implantation of the positron remoderation processes. Since the elevation does not influence other beam parameters, the brightness and the time structure of the positron beam is conserved. This device allows also keeping both, the source and the sample, at the same electrical potential.

## Large-scale structural change of a FRET-based biosensor investigated by SAXS and SANS

ID 279

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Genetically encoded FRET-based biosensors have been developed as a tool to quantify metabolites in living cells. The sensors consist of a central metabolite binding protein and flanking fluorescent GFP proteins affixed by different linker sequences.[1] Fluorescence measurements are sensitive either to distance or orientation changes of GFP domains as response to glucose binding. They were used to determine a change in energy transfer upon glucose binding. SEC(size exclusion chromatography)-SAXS measurements have been performed to investigate, if either large-scale structural change of the GRP positions or relative orientation changes occur as response to glucose binding. Based on the measured SAXS curves we have performed rigid body modelling of the GFP domains. These structures fit with the behaviour of the sensor expected due to fluorescent measurements. In order to gain a better understanding of the sensor under macromolecular crowding conditions mimicking the cellular state SANS experiments have been performed where the sensor was surrounded by contrast matched partially deuterated PEG 6kDa in different concentrations.

[1] V. Steffen et al, 2016, Sensors, vol. 16, no. 10, p. 1604

## Residual Stress Characterization of Friction Surfacing Coated AA2024 Plates

ID 290

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Friction Surfacing (FS) is a solid-state process, which allows deposition welding at temperatures below the melting range. It can be used, e.g., for property enhancement or repair purposes. The technique uses a consumable stud that is rotated and pressed on a substrate surface while moved forward. The combination of materials used for stud and substrate defines the windows of process parameters: stud rotational and translational speeds and axial force. The rotational and translational stud speeds together with substrate geometry determine the temperature regime during deposition and the geometry of the coating. The focus of this investigation was the effect changing FS translational stud speed and substrate thickness have on the residual stress (RS) state of the AA2024 plates. Combined neutron and synchrotron diffraction analysis were used to determine the RS fields induced by the deposition process in both substrate and coating. The obtained results allow drawing conclusions on which FS parameters have most important effects on the RS state. Moreover, an estimative model of the RS formation during and after FS deposition can be developed.



## Structural Investigations of a Model System for Multiple Sclerosis

ID 291

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The myelin sheath is the membrane responsible for rapid signal transport in neurons. In multiple sclerosis, this membrane is damaged leading to neuron conduction failure. Myelin Basic Protein (MBP) which is the major structure protein of the myelin sheath has been proven to be an important factor in stabilizing the myelin sheath. In recent publications, a different lipid composition has been found for native and diseased membranes. Based on these results, we built membranes mimicking native and diseased myelin sheath lipid composition. In this model system, we want to investigate the interaction mechanism of MBP with the different membranes. Here, we present neutron reflectometry (MARIA of JCNS at MLZ Garching) and small angle neutron scattering (SANS) (KWS-2 of JCNS at MLZ Garching) data which show structural differences of native and diseased membranes. Furthermore, the injection of MBP to native and diseased membranes is shown to lead to a major change of the membrane structure.



## Moisture-Induced Degradation in Perovskite-Based Photovoltaics

ID 294

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Hybrid organic-inorganic halide perovskites  $\text{CH}_3\text{NH}_3\text{PbX}_3$  ( $X = \text{I}; \text{Br}; \text{Cl}$ ) are promising novel materials for the application in future photovoltaics (PVs), with a tunable absorption characteristic and efficiencies comparable to silicon. Since solution-based fabrication techniques and the use of flexible substrates are possible, perovskite PVs are potentially low-cost and could find application in new fields, where conventional silicon is unfeasible. However, perovskite is sensitive to moisture, which seems to primarily drive the degradation of the crystal structure. Some of the typically used contact layers also show long-term degradation. Achieving long-term stability of perovskite and of its contact layers would therefore open a wide range for future applications. We will analyze the moisture-induced morphological changes of typically employed contact layer materials and in mixed halide perovskite with X-ray diffraction techniques, UV/VIS spectroscopy, scanning electron microscopy, and photoluminescence. Especially grazing incidence scattering techniques allow us to trace the degradation across the sample on an area relevant for devices. The characterization and understanding of the degradation process will help us to select suitable materials with an improved long-term stability. Designs with a promising stability will be subjected to in-situ measurements, to directly assess the nano-morphological changes which drive the degradation of perovskite PVs.

## Sharper X-ray vision through aberration-corrected optics

ID 295

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The high brilliance of modern synchrotron radiation and X-ray free-electron laser sources allows studying the structure and dynamics of matter on relevant lengths and time scales. Creating small and intense X-ray beams is crucial to confine the beam and concentrate the radiation onto the sample. Ideally, this would require diffraction-limited X-ray optics with high numerical aperture (NA) that are also stable in the intense X-ray beam of current and future facilities. While the short X-ray wavelength allows creating foci down to a few nanometers and below in theory, it is this short wavelength that puts stringent requirements on X-ray optics and their metrology. Both are limited by today's technology. To overcome this barrier a corrective phase plate can be employed that rectifies any aberrations if wavefront errors are known in detail. Here, I'll show the at wavelength measurement of aberrations in beryllium compound refractive lenses using ptychography and present their elimination by tailor-made corrective phase plates based on these data. The optical system composed of the original lens and the phase plate achieves diffraction-limited performance with high NA. The approach is not only limited to refractive optics, but can correct diffractive and reflective optics beyond current manufacturing limitations as well. In addition, also existing optical systems can be upgraded due to the compact size and easy implementation of the phase plate.

## Internal stress in electrical steel sheets – Effects and Applications

ID 296

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Electrical steel sheets are used in transformers as well as in electrical motors to guide the magnetic field. The efficiency of an electrical steel sheet strongly depends on the amount of energy lost during the reversal of magnetization, which is dependent on the mobility of the magnetic domains. The mobility of the magnetic domains is mainly influenced by internal stress caused during the manufacturing process. [1],[2]. To probe the magnetic domain constellation in bulk samples of technically relevant dimensions neutron grating interferometry (nGI) is the technique of choice, as it allows to probe the bulk local magnetic properties, which is not possible with most other techniques. nGI provides information about the amount of ultra-small-angle-neutron scattering (USANS) inside a sample [3]. The resulting image (DFI) is sensitive to the distribution of magnetic domain walls, which serve as possible scattering centers. Hence the DFI signal is related to the local distribution and size of magnetic domains inside a sample. We will show how internal stress affects the local hysteresis of an electrical steel sheet, causing a degradation of the magnetic domain mobility. Furthermore, we will show how an intended use of this effect, due to imprinting on the electrical steel sheets can increase the efficiency of an electrical engine.

[1] H. Weiss et al., pending (2018)

[2] A. Moses, IEEE Trans. Magn, Vol. 15, 1575-1579 (1979)

[3] C. Grünzweig, PhD thesis (2009)

## Implanter applications of polyatomic ions from a high current liquid metal alloy ion source

ID 298

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High current liquid metal ion sources are versatile investigated and found their first application as field emission electric propulsion (FEEP) thrusters in space technology [1]. Due to the available ion current in the  $\mu\text{A}$ -range such kind of sources are also well suited for broad ion beam technology. LMAIS are also nearly the only type of ion sources delivering polyatomic ions from about half of the periodic table of elements [2]. Surface patterning based on self-organized nano-structures on e.g. semiconductor materials formed by heavy mono - or polyatomic ion irradiation from liquid metal alloy ion sources (LMAIS) is a very promising technique demonstrated using a focused ion beam (FIB) equipment [3]. To overcome the lack of only very small treated areas by applying a FIB working with such sources, the technology taken from space propulsion systems was transferred into a large single-end ion implanter. The main component is an ion beam injector containing the high current LMAIS combined with suited ion optics allocating a high current nearly parallel ion beam of a few mm in diameter. Different kinds of LMAIS (needle type, porous emitter, capillary type) are presented and characterized with respect to their performance. The ion beam injector design is specified as well as the implementation of this module into a commercial 200 kV high current ion implanter (Danfysik 1090) operating at the HZDR Ion Beam Center. Large area surface modification of Ge using polyatomic  $\text{Bi}_2^+$  ions at room temperature emitted from a GaBi capillary LMAIS will be presented [4] and compared with the results of FIB technology.

[1] M. Tajmar and B. Jang, New materials and processes for field emission ion and electron Emitters, CEAS Space J. 4 (2013) 47.

[2] L. Bischoff, P. Mazarov, L. Bruchhaus, and J. Gierak, Liquid Metal Alloy Ion Sources - An Alternative for Focused Ion Beam Technology, Appl. Phys. Rev. 3 (2016) 021101.

[3] R. Böttger, L. Bischoff, K.-H. Heinig, W. Pilz, and B. Schmidt, From sponge to dot arrays on (100)Ge by increasing the energy of ion impacts, J. Vac. Sci. Technol. B 30 (2012) 06FF12.

[4] W. Pilz, P. Laufer, M. Tajmar, R. Böttger, and L. Bischoff, Polyatomic Ions from Liquid Metal Ion Source driven High Current Ion Implanter, Rev. Sci. Instrum. 88 (2017)123302.

## Exploring dynamic structures of bionanocages by SRCD

ID 299

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Given the vast array of natural synthesized proteins, the degree of folding, mechanism, location of assembly and size of the protein varies greatly. Amid this multitude of proteins, the ferritin family of proteins, considered bionanocages can be classified by size. The DNA-binding proteins from starved cells (DPS) are under 10 nm in diameter and part of this family. These bionanocages are characterized by their globular shape with a hollow interior cavity. DPSs are involved in several metabolic pathways such as detoxification, iron sequestration, oxidative stress and radiation damage prevention. A specially interesting ability of the cavity is the possibility to, without bioengineering, synthesize different types of nanoparticles, forming specific metal cores. Another interesting possibility is the use of the hollow cavity as a drug delivery system. Given the wide array of biotechnology uses that DPS bionanocages can achieve, it is important to have an understanding of the hollow cavity dynamic interactions during synthesis of different compounds. In order to gain a deep insight into the dynamics of the denaturation of DPS, Synchrotron Radiation Circular Dichroism (SRCD) measurements were performed at ASTRID2, Aarhus University. This allowed the assessment of melting temperatures and secondary structure features providing insights on cage assembly/disassembly. This information clarifies potential ways on how one can control the bionanocage for biotechnological applications.

## Fast Detectors and Electronics for Nuclear Resonant Scattering of Synchrotron Radiation

ID 302

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In nuclear resonant scattering (NRS) experiments the nuclear decay following the excitation of the sample by a synchrotron radiation pulse is monitored by fast avalanche photo diodes (APD) [1]. In this presentation we focus on recent developments on a 16 element APD detector, the application of a fast time to digital converter (TDC) [2] for enhanced pulse detection, and a fast digitizer for time resolved pulse shape analysis. The 16 element APD detector consists of 16 single Hamamatsu APDs (type S5444LC, 3 mm diameter and 30  $\mu\text{m}$  thickness), yielding a time resolution of 0.3 ns each. By inclining and stacking all modules one is able to accept variable beam size, the beam intensity is distributed on all APDs, resulting in a reduced load on each APD and the performance especially at early times after the intense prompt pulse is significantly improved. In this arrangement we achieved an efficiency of about 15% and a time resolution below 0.8 ns at a photon energy as high as 73 keV at the  $^{193}\text{Ir}$  resonance. Classical NIM-based timing electronics requires a veto region up to 8 ns after the prompt pulse. The TDC (FAST Comtec MCS6A, up to 5 input channels) can operate veto-free, with 100 ps time resolution up to 8 MHz event rate. It allows fitting of data already 2.5 ns after the prompt pulse. Moreover, the TDC provides the capability to count multiple events per excitation, a feature, which is, e.g., important for NRS experiments at free electron lasers. The fast digitizer based on commercial electronics from SP devices [3] allows for time resolved pulse height and shape analysis with a sampling rate of 4 GHz. This allows one to time and count multiple events per excitation, and in Nuclear Inelastic Scattering the digitizer provides the possibility to separate the time dependencies of the 6.4 keV x-ray fluorescence and 14.4 keV gamma fluorescence recorded simultaneously.

1) A.Q.R. Baron, S. Kishimoto, J. Morse, J.-M. Rigal, JSR 13, 131 (2000)

2) <https://www.fastcomtec.com/>

3) <https://www.spdevices.com/>

## Transverse Free-Electron Target for the Heavy-Ion Storage Ring CRYRING@ESR

ID 306

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It is planned to install a ribbon-shaped high-density free-electron target in the experimental section of the presently being commissioned storage ring CRYRING@ESR of the upcoming Facility for Anti-proton and Ion Research (FAIR) [M. Lestinsky et al., Eur. Phys. J Spec. Top. 225, 797 (2016)]. Electron beam and circulating stored ion beam interact under an angle of  $90^\circ$ . Recently, we have developed a new versatile electron gun with electron energies up to 12.5 keV which is optimized for the storage ring environment as well as for photon spectroscopy. This gun is presently built as well as the entire target station. In this contribution we present the physics opportunities of such a free-electron target at a heavy-ion storage ring and give an overview about the present status of the project.

## GISAXS investigation of highly ordered nanostructures

ID 307

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GISAXS is a powerful tool to provide information about the size, shape and structural ordering of nanostructures. For evaluation of the experimental 2D patterns an appropriate software is needed. In this work we study nanostructures with well defined form and structure factors which will be used as reference samples. The main aim of the studies is to prepare the BornAgain software package to perform fits of 3D models directly to the experimental 2D patterns. Appropriate reference samples were prepared via electron beam lithography in cooperation with MPI (Erlangen). Arrays of cylinders with definite radii, heights and distances were etched in silicon wafers. These arrays of cylinders in turn were arranged in a field with an overall area of about one square millimetre. From such samples 2D interference patterns of the real space structures with periodicities well above one micrometre could be well resolved by lab based GISAXS studies. We present simulations and fits of our measurements using the BornAgain software package including studies of more complex systems as printed active layers of thin film solar cells, nanoparticulate layers on silicon, and 2D ordered aluminium oxide tubes.

## Internal dynamics of different folding intermediates of apo-myoglobin

ID 309

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During protein folding different intermediate states occur with varying content of secondary structural elements. Regions lacking well-defined structure exist as well in unfolded and partially folded systems. In order to characterize their dynamics, we studied apo-myoglobin in different folding states [1,2,3]. We investigated internal dynamics of its unfolded form on a time-scale up to several hundred nanoseconds and in the nanometer length-scale using neutron spin echo spectroscopy (NSE) at the instrument SNS-NSE in Oak Ridge[4]. We measured the folded protein with NSE on the same time scales for comparison at the J-NSE "Phoenix" at MLZ. Aggregation state and center of mass diffusion were monitored in both cases in parallel with dynamic light scattering. Information on the form and structure factor was obtained by small angle neutron scattering (SANS) at KWS-2 at MLZ. Whereas the dynamics of the folded protein is dominated by center of mass diffusion, our first NSE data of the unfolded protein state shows a polymer-like behavior. This indicates that powerful polymer models well-established in the field of soft matter may be used to describe such protein systems.

[1] J. Phys. Chem. B, 2015, 119 (1), 72

[2] J Mol. Biol., 1996, 263(4), 531

[3] J. Am. Chem. Soc., 2014, 136 (19), 6987

[4] Richter D. et al., 2005, Neutron Spin Echo in Polymer Systems. Advances in Polymer Science, vol 174. Springer, Berlin, Heidelberg

**Reference-free GIXRF-XRR based characterization of nanolayers and nanostructures**

ID 310

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In most cases, bulk-type reference-materials do not provide optimal calibration schemes for the analysis of nanomaterials as e.g. surface and interface contributions may differ from bulk. Also, spatial inhomogeneities may exist at the nanoscale or the response of the analytical method may not be linear when going from bulk to the nanoscale. Thus, the availability of suited nanoscale reference materials is drastically lower than the current demand. The reference-free X-ray fluorescence (XRF) technique of PTB can address this disparity as it enables an SI traceable quantitative characterization of nanomaterials without the need for reference or calibration standards. This opens a route for the XRF based qualification of calibration samples. As a first example, we use physical vapor deposition techniques for the development of layer-like reference samples with very low mass depositions. Different reference samples were fabricated for application in XRF and total-reflection XRF analysis. They were quantitatively characterized using synchrotron radiation employing PTB's reference-free XRF approach. In a second example, we work on the development of nanostructures as calibration samples. Several lithographic 2D and 3D nanostructures have been fabricated at PTB using e-beam lithography and were characterized using the reference-free grazing incidence XRF methodology of PTB. Here, an advanced and novel calculation scheme for the intensity distributions within the X-ray standing wave field (XSW) is required. In addition to the traceable quantification of elemental mass depositions, this allows for a determination of in-depth elemental distributions and the dimensional properties of the nanostructures.



## Efficient X-ray optics for spectroscopy of highly charged ions

ID 311

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Spectroscopy of few electron heavy ions is a unique testbed for both atomic physics, especially the test of quantum electrodynamics in strong fields, as well as for tackling astrophysical questions. The energy resolution of such measurements is limited by the use of standard semiconductor detectors. Currently, there are two ways to improve the energy resolution under investigation: on one side the use of crystal spectrometers, and on the other side, the use of magnetic metallic microcalorimeters. These new detectors could have energy resolution close to 1 eV below 10 keV and around 10 eV in the range below 100 keV. Their drawback is a small detection area and thus small covered solid angle. The detection efficiency of the microcalorimeters could be improved by the use of x-ray optics based on high aperture mosaic crystals, which are used to transport radiation from the source to the detector. We developed, build and tested optics based on pyrolytic graphite and lithiumfluoride that could increase photon flux on the detector by up to two orders of magnitude in the energy range between 6 and 30 keV.

## Neutron Depth Profiling with Highest Luminosity Pulsed Beams

ID 312

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One major limitation in neutron depth profiling is given by the signal to background ratio (S/B) especially using thick sample material or samples with high amount of absorbing elements. A high neutron flux for fast and time resolved measurements introduces typically a fast increase of beta activity in the sample material and dilutes the S/B ratio. Shielding techniques for beta particles and delayed gamma rays cannot be applied in NDP. Also the detector active volumes have already reached physical limitations. The most promising way out is a direct correlation of impinging neutron with particle emission after the absorption. A major step toward such a scheme is provided by the use of pulsed neutron beams. With a beam duty cycle of 4% for example at the ESS we expect the random background being suppressed by at least one order of magnitude. This would especially allow us to study material compositions with heavy absorbing elements as metals or strongly activating components and opens up a complete new measurement scheme in NDP. First ideas and necessary conditions will be presented in this contribution.



## P66 beamline for time-resolved luminescence experiments under VUV excitation at PETRA synchrotron

ID 313

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The first works on the new beamline P66 design has been started 5 years ago as a part of PETRA III extension project [1]. At the very beginning of the project the main idea was to relocate very successful experimental station for time and spectrally resolved luminescence investigations from DORIS to the PETRA synchrotron source. The efficiency of the unique station SUPERLUMI at DORIS [2] was very high. The efficiency of the new P66 station is expected to be at least at the same level. The P66 will operate in the UV- VUV spectral region with pulsed synchrotron radiation excitation. A comparable time resolution in nanosecond range on one hand as well as an increasing number of users with high publications output on the other hand outlined a strong motivation for the future of the P66 at PETRA synchrotron. On the later stage of the project the idea came to consideration, to add laser for two photon experiments. The goal is to establish innovative new pump-probe capabilities to close the gap in time resolution between nanoseconds and femtoseconds. The final version of the layout of the P66, the parameters calculated for the optical elements, twophoton experiment details and timelines for construction site will be presented. Major changes of the beamline setup and electronics upgrade should make experiments more convenient and efficient for more than 20 scientific groups from all over the world.

[1] <http://petra3-extension.desy.de/>

[2] G. Zimmerer, Radiation Meas. 42, (2007) pp. 859-864

## Study of Magnetic Dumbbell nanoparticles using advanced scattering techniques

ID 317

**NANDAKUMARAN, Nileena**

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Magnetic dumbbells consist of a noble metal seed linked to a magnetite nanoparticle. Self-assembly of nanoparticles in general, is of interest due to its broad range of applications in material science and biomedical engineering. Parameters that affect self-assembly of nanoparticles include particle size, thickness of surfactant and concentration but additional parameters such as composition of the seed, the interface between seed and magnetite, shape anisotropy and magnetic structure also influence self-assembly of dumbbells induced by magnetic fields. In order to understand the parameters that influence self-assembly of dumbbells, we must first investigate self-assembly in single phase magnetite nanoparticles. Our studies on magnetic nanoparticles with 16nm and 27nm diameter from small-angle neutron scattering reveal a profound size effect on self-assembly. These measurements will act as a reference for future studies of dumbbells with comparable parameters. We will discuss our assessment of the complete magnetic and nuclear cross-sections of the dispersed and self-assembled structures, determined from Polarisation Analysis, Polarised and unpolarised Small-Angle Neutron Scattering (SANS). SAXS on single phase nanoparticles and dumbbell nanoparticles with and without field are reported to complement the results of neutron scattering experiments. Further, magnetisation measurements on these samples reveal their blocking temperature and interesting magnetic behaviour.



## Generation of high-density positron pulses at NEPOMUC for production of an electron-positron plasma

ID 318

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A pair plasma produced of electrons and positrons will enable novel studies of many facets of fundamental plasma physics, in addition to being of great astrophysical relevance. The main bottleneck in producing this exotic system in a laboratory is the command over a sufficient number of positrons. We are therefore using the NEPOMUC positron beam line at FRM II for our project. For our plasma experiments, the beam is injected into a toroidal magnetic confinement volume where it will be mixed with electrons. The general layout of this experiment, and an update about its status will be presented. In order to provide a sufficiently high positron flux to our experiment, we have engaged in a series of systematic studies of the NEPOMUC beam properties, results of which will be detailed. We have succeeded in guiding the primary beam (which has a higher intensity than the commonly used remoderated beam) to the open beam port and further injecting it into our plasma device. We expect to achieve further substantial improvement by temporally bunching the positron flux. Towards this end, we will install at NEPOMUC a buffer gas trap, which can store up to 108 positrons and deliver them within 10<sup>-7</sup> seconds to an experiment. We expect that this will enable a number of novel experiments in diverse areas of positron physics.

### 3D Material Characterization by Laminographic Imaging: Status and Prospects

ID 328

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Laminography enables non-destructive 3D imaging, achieving high resolutions with multiple contrasts for selected regions of interest even for large and laterally extended samples exceeding the view field. We continuously progress in the theoretical description of the methodology, in the construction of dedicated instrumentation, the measurement procedures, and the algorithms for data analysis. With the recent development of X-ray Diffraction Laminography (XDL) within the German-Russian BMBF project STROBOS-CODE, for example, laminography has been extended with Bragg-diffraction contrast [1]. This enables the 3D characterization of crystal defects like complex dislocation networks with a few micrometer spatial resolution, within large crystal volumes as typical e.g. for technology-relevant semiconductor wafers. By the correlative analysis of XDL with data obtained by means of complementary techniques, we could gain new insight into the onset of thermal slip in silicon wafers under processing-relevant conditions [2, 3].

[1] Hänschke et al., APL 101, 244103 (2012).

[2] Hänschke et al., PRL 119, 215504 (2017).

[3] Redmond, MRS Bulletin 43, 11 (2018).

## Upgrades at SPHERES

ID 330

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The SPectrometer for High Energy RESolution (SPHERES) at MLZ is a third generation backscattering spectrometer with focusing optics and phase-space transform (PST) chopper. It provides high energy resolution with a good signal-to-noise ratio [J. Wuttke et al., Rev. Sci. Instrum. 2012]. Different components of the instrument have been upgraded to further improve the instrument performance. Some recent years ago the PST chopper has been renewed. The new more compact one-wing chopper can be operated with the desired frequency with a crystal speed of 225m/s close to the optimum velocity for the phase space transformation. Together with the exchange of the chopper also the graphite deflector crystals on its circumference were replaced with ones of a higher reflectivity and mosaicity. Thanks to the increased velocity and the better deflector crystals, the intensity in most detectors had been doubled. Just recently the focusing neutron guide has been replaced with an elliptic guide. It had been optimized based on simulations, which also considered the new PST chopper. With the new elliptic guide another intensity gain at the sample position of about 30% was obtained. Together with the exchange of the focusing guide also a new background chopper has been installed about 2m upstream of the PST chopper to further reduce background. This will then also allow for a high signal-to-noise setup by eliminating every second pulse, albeit at the cost of intensity.



## Phase Retrieval for X-Ray Near-field Holography beyond Linearisation

ID 334

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X-ray near-field holography (NFH) is a propagation based coherent phase contrast imaging technique which does not rely on an image forming optic. Implementing NFH in a divergent beam created by nano focusing optics allows the application for microscopy. The magnification and field of view are straightforwardly controlled by geometrical parameters. This allows the easy measurement of overview and zoom images of the same specimen. However, this image has to be obtained by carrying out the phase retrieval on the measurements. For high resolution synchrotron experiments, phase retrieval is largely based on the single step reconstruction using the contrast transfer function approach, as introduced almost twenty years ago. Notwithstanding its tremendous merits, this scheme makes stringent assumptions on the optical properties of the object, requiring in particular a weakly varying phase. In this contribution we show how significant the loss in image quality becomes, if these assumption are violated, and how phase retrieval can easily be improved by a simple scheme of alternating projections. Importantly, the approach demonstrated here uses the same input data and constraint sets as the conventional CTF-based phase retrieval, and is particular well suited for the holographic regime.

## Neutron structure analysis of NADH cytochrome b5 reductase

ID 335

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Redox proteins functions in many biological processes, such as metabolism, photosynthesis, respiration. The chemical reactions of redox proteins proceed with small structural changes involved in hydrogen atoms and/or valence electrons. Thus, high-resolution structure analysis revealing these small structural changes is necessary for understanding molecular mechanisms of redox reactions. NADH cytochrome b5 reductase (b5R) catalyzes the electron transfer from two electron carriers of NADH to one electron carrier of cytochrome b5 (b5). High-resolution X-ray structure analyses have been reported for b5R [1-2] and b5 [3] from porcine liver. Recently, we have performed high-resolution neutron crystal structure analyses of b5R. We succeeded in data collection of b5R (oxidized form) at high resolutions, 1.40 Å (at iBIX in J-PARC) and 1.45 Å (at BIODIFF in FRM-II), under cryogenic conditions. The logarithmic plot of average diffraction intensities against resolution shows gradual decrease from low to high resolutions in the BIODIFF data, in contrast the plot shows a flat line at the high resolution side in the iBIX data. We have performed the correction of diffraction intensities for the iBIX data using the BIODIFF data.

[1] M. Yamada et al. J. Mol. Biol. 2013, 425, 4295-4306. [2] K. Takaba et al. Sci Rep. 2017, 7, 43162.

[3] Y. Hirano et al., Acta Crystallogr. D 2015, 71, 1572-1581

## Cryptoferromagnetic-like effect in Gd/Nb superlattice

ID 340

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The proximity effects in superconducting/ferromagnetic (S/F) systems are attracting nowadays great attention in basic and applied research due to the presence of unique properties. Typically the S/F systems consisted of F layers with rather strong exchange energy (100-1000K) in contact with much weaker (10-100K) superconductors. In this sense it is straightforward to expect strong influence of ferromagnetic ordering on superconductivity, starting from trivial pair-breaking to such intriguing effects like Larkin-Ovchinnikov-Fulde-Ferell state,  $\pi$ -type Josephson junctions, triplet superconductivity and more. On the other hand, influence of superconductivity on ferromagnetic state is less expected due to the above mentioned strong difference in energies. One of such effects is cryptoferromagnetism. Essence of the effect in creation of domain structure with domain size less than correlation length of superconductivity  $196\xi_F$  below the superconducting transition temperature  $T_c$ . Effect is predicted for the S/F systems with weakened ferromagnetic energy and enhanced superconducting energy. In this talk we present results of polarized neutron investigations of  $[\text{Gd}(dF)/\text{Nb}(25\text{nm})]_{12}$  superlattices with  $dF = 1-5\text{nm}$ . We have observed suppression of magnetic ordering of Gd layers in superlattices with  $dF < 196\xi_F$ . Relation of the experimental observation to cryptoferromagnetism is discussed.

## SI traceable characterization of nanomaterials by X-ray spectrometry

ID 342

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The development of new materials and the assessment of nanomaterials require the correlation of the materials' functionality or toxicity with their chemical and physical properties. To probe these properties, analytical methods that are both sensitive and selective at the nano- and microscales are required. The reliability of most analytical methods is based on the availability of reference materials or calibration samples, the spatial elemental composition of which has to be as similar as possible to the matrix of the specimens of interest. However, there is a drastic lack of reference materials in particular at the nanoscale. PTB addresses this challenge by means of a bottom-up X-ray analytical method where all instrumental and experimental parameters are determined with known contributions to the uncertainty of the analytical results. This first-principle based approach does not require any reference materials but a complete characterization of the analytical instruments' characteristics and, in addition, knowledge on the X-ray fundamental parameters related to the elements composing the sample. SR based X-ray spectrometric methods allow for the variation of the analytical sensitivity, selectivity, and information depth needed to effectively reveal the spatial, elemental, and chemical specimen parameters of interest. Examples of particle characterization, interfacial speciation, elemental depth profiling, as well as layer composition and thickness characterizations in advanced materials will be given. Recent instrumental achievements provide access to liquids, liquid-solid interfaces as well as the in-situ and operando elemental analysis and chemical speciation of nanoscaled battery materials. X-ray spectrometry under grazing incidence is capable to reveal analytical and dimensional information from layered systems and particles deposited on surfaces.

*(References shortened by editors)*

**DMPC model membrane structure and dynamics in the presence of the saponin aescin**

ID 344

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Vesicle shape and bilayer parameters of 1,2-dimyristoyl-sn-glycero-3-phosphocholine based vesicles are studied by small-angle X-ray (SAXS) and small-angle neutron (SANS) scattering in the presence of the natural surfactant (saponin) aescin [1]. We confirm successful incorporation of aescin molecules by analysis of the radii of gyration of the vesicles which we use as model systems. Furthermore, we study the impact of aescin incorporation on bilayer thickness parameters from the neutron and X-ray perspective. Additionally, the bending elasticity of these vesicle bilayers is studied in the presence of aescin. Neutron spin-echo spectroscopy (NSE) allows to detect subtle changes in the dynamics and mechanics of lipid membranes. Changes of the bending elasticity are detectable at temperatures below and above the main phase transition temperature of the lipid. The impact of aescin is much more significant below . It has been found that below the phase transition temperature the addition of aescin to the vesicles softens the bilayer. Above the phase transition temperature the value of bending elastic constant increases with increasing aescin content and the bilayer becomes more rigid. Altogether, we demonstrate by analysis of the structure and dynamics of the vesicles that the impact of aescin strongly depends on the lipid state [2].

[1]Sreij, R., Dargel, C., Moleiro, L., Monroy, F., & Hellweg, T.; *Langmuir* (2017), 33 (43), 12351- 12361.

[2] Sreij, R., Dargel, C., Geisler, P., Hertle, Y., Radulescu, A., Pasini, S., Perez, J., Moleiro, L. H. & Hellweg, T., *Physical Chemistry Chemical Physics* (2018), 20, 9070-9083

## In situ triaxial deformation experiments on a sandstone sample for strain investigation at the neutron time-of-flight diffractometer EPSILON

ID 351

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The investigation of uniaxial and triaxial stress states in geological samples serves to a better understanding of the rock behaviour. The application of neutron time-of-flight diffraction allows the study of applied and residual strain of bulk rocks. The neutron time-of-flight strain diffractometer EPSILON at IBR-2M is equipped with a new triaxial pressure device "TRIXI" to investigate polycrystalline rock samples at pressures, representing upper crustal reservoir conditions. Axial pressure up to 150 MPa, confining pressure up to 70 MPa and additional pore pressure up to 70 MPa can be operated, independently. Strain analysis up to d-spacing of 5.3 Å are achievable. We present the first results using a porous sandstone for in situ strain experiments. This sandstone is considered as reservoir analogue for most of the North German and Dutch gas fields. We compare the results with modelled stress distributions and theoretical expected results. This new device may be used to address questions on reservoir properties under changing pore pressures, like injection of waste water, geothermal use or production of gas, water, and oil.

## Structural investigation of Li<sub>6</sub>PS<sub>5</sub>Cl for applications in all-solid-state batteries

ID 359

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In the recent years the application of lithium-ion-batteries has attracted a greater interest for large scale applications such as electric vehicles (EV). Liquid batteries are already used and commercialized for such applications. This battery type has higher safety concerns in terms of leakages and flammability, especially in case of a crash. A good alternative with lower risks for drivers and passengers are all-solid-state batteries. Typical materials are polymers, oxides and sulfides. Polymers show a conductivity up to  $10^{-3}$  S/cm at about 80°C, whereas oxides have similar conductivities at room temperature. Sulfidic materials can reach up to  $10^{-2}$  S/cm which is similar or even better than liquid batteries. One typical crystalline sulfidic material is Li<sub>6</sub>PS<sub>5</sub>Cl with an argyrodite like structure. A better understanding of its structure, structural changes and its impact on lithium ion conduction pathways over a large temperature range is necessary. An intensive investigation on Li<sub>6</sub>PS<sub>5</sub>Cl with Synchrotron and Neutron diffraction methods will be presented in this work.

## SKADI - Small-Angle Neutrons Scattering at the ESS

ID 360

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The Small-K Advanced Diffractometer (SKADI) is a small-angle neutron scattering (SANS) instrument to be built at the European Spallation Source (ESS). It will enable scientists to perform experiments on questions requiring to investigate the microscopic structure of samples over a wide range of length scales and fast kinetic experiments. SKADI's design targets scientific areas of smart materials, biological and pharmaceutical research, magnetic materials and materials for energy storage, as well as experiments on nanomaterials and nanocomposites of colloidal systems. The sample area (3×3 m<sup>2</sup>) and sample environment will also increase the applicability of the research performed on SKADI by accommodating in-situ and realworld sample environments. Additionally, by the open design, custom sample environments can be incorporated easily. The generic mounting system will also allow off-instrument preparation of the sample environment, facilitating the change-over procedure between different experiments. The additional features of SKADI are a wide dynamic Q-range ( $10^{-4} \text{ \AA}^{-1} \leq Q \leq 1 \text{ \AA}^{-1}$ ) for single shot experiments, polarization, a tunable (time-of-flight based) wavelength resolution down to  $\Delta\lambda/\lambda = 1\%$  combined with collimation settings for 20, 14 and 8 m and aperture sizes between 0 and 30 mm. The usable wavelength band for standard operation is 5 Å, a pulse-skipping mode allows for a 10 Å wavelength band. The available flux at sample position will be several times 10<sup>8</sup> neutrons/s cm<sup>2</sup>.

## In-situ neutron scattering study of adsorption induced structural deformation in CAU metal organic frameworks – can structural contraction increase hydrogen uptake?

ID 362

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Understanding the molecular mechanism of adsorption in porous materials is important for a broad range of modern technologies starting from energy storage and conduction to heterogeneous catalysis and to the development of the novel drug carriers. Recent theoretical studies hint at adsorption induced structural changes and the onset of stress in porous materials contradicting broadly accepted assumption that the structure of the porous materials does not change during the sorption process. In order to understand the origin of such changes and their impact of the adsorption behavior we have studied hydrogen sorption in CAU-1 and CAU-8 metal organic frameworks. The structures of CAU-1 and CAU-8 consist from 3D pore network and 1D confining channels respectively [2],[3]. Built from aluminum polyhedra with fully coordinated metal ions CAU-1 and CAU-8 provide an environment with similar pore wall interactions for guest molecules. This allow us to systematically follow the intermolecular interactions as a function of the pore geometry. Using in-situ neutron scattering techniques we observe a substantial structural contraction at the initial stages of adsorption in both materials, which originate from interactions between the hydrogen and organic linkers. In CAU-1 such contraction triggers the rearrangements of confined molecules and, surprisingly, to the formation of new occupational positions, increasing the hydrogen uptake [4]. In 1D channel system CAU-8 the adsorption of hydrogen on the linkers is followed by the formation of the hydrogen chains close to the channel center and, as a result, to the structural relaxation of the host framework. In both systems we see novel structural arrangements of confined hydrogen at nanoscale.

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[2]. F. Hinterholzinger N. Stock et al, Phys. Chem. Chem. Phys., 2010, 12, 4515–4520

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[4]. M. C. Schlegel, M. Russina et al, PCCP, 2016, DOI: 10.1039/C6CP05310F

## Thermally induced cation re-ordering in activated Li and Mn-rich layered oxide Li-ion cathode materials

ID 365

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Layered transition metal oxides such as the 'Li- and Mn-rich' layered oxides, formed as the composites between  $\text{Li}_2\text{MnO}_3$  and  $\text{LiMO}_2$  ( $M = \text{Ni, Co, Mn}$ ) (NCM) are promising candidates for next generation Li-ion battery cathodes that offer high reversible capacities ( $> 250 \text{ mAh/g}$ ) and higher safety together with reduced costs. For an economic and efficient operation as cathode materials in electrified vehicles for example, knowledge about mechanisms of degradation in order to optimize these materials for longer lifetime is mandatory. So far, a gradual decrease of the energy density during electrochemical cycling called 'voltage and capacity fade' is a major drawback of this material class. A local cation rearrangement towards a cubic  $\text{LiMn}_2\text{O}_4$  spinel-like symmetry during de-/lithiation is supposed to cause this energy decay in many publications. In order to study the thermodynamically preferred cation arrangement in dependency of the lithiation grade, the structural transformation was thermally induced and investigated by global synchrotron diffraction as well as local nuclear magnetic resonance and X-ray absorption techniques. Furthermore, the structural characteristics of the artificially fatigued samples were compared with conventional highly cycled electrodes and finally correlated with their electrochemical properties

## Particle Detectors with Arduino-based Frontend Electronics

ID 367

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With the Arduino open hardware electronics platform microcontrollers have become a comparably easy-to-use tool for rapid prototyping and implementing innovative solutions. Their stability especially qualifies them to be used for slow control units. Yet, running at 16 MHz, the capabilities can be extended to data taking and signal analysis at decent rates. Such devices in combination with dedicated frontend electronics can offer low cost alternatives for student projects and independently operating small-scale instrumentation. We present two projects: the readout of proportional counters using helium-3 and boron-10 and a trigger unit for reading out scintillators or wavelength shifting fibers with Silicon Photomultipliers. The nCatcher combines commercially available analog electronics and the Arduino nano enabling pulse shape analysis for proportional counters. The frontend integrates and shapes pulses to microseconds in order to use the Arduino's signal analysis capabilities - time over threshold measurement and a 10-bit analog to digital converter. Combining these two parameters allows for effective discrimination between different radiation types by estimating the total deposited energy ( $E$ ) of incoming radiation as well as the mean energy loss per distance ( $dE/dx$ ). This makes the device suitable for low to medium rate environments, where a good signal noise is a crucial. With the SiPMTrigger we have realized a small scale design for triggering or vetoing. It consists of a custom mixed signal frontend board featuring signal amplification, discrimination and a coincidence unit. An Arduino MEGA digitally adjusts the thresholds of both channels and measures the trigger rate up to 200 kHz.

## Development of a high-brilliance accelerator driven neutron source: Simulations for NOVA ERA

ID 370

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The High Brilliance neutron Source (HBS) project [1] aims for the development of compact, scalable, accelerator driven neutron sources. These are needed to facilitate the installation of a network of low to medium flux sources in Europe and close the developing supply gap for science and industry. The NOVA ERA (Neutrons Obtained Via Accelerator for Education and Research Activities) [2] branch of the HBS project focusses on a low flux solution to be operated at universities, research institutes or industry laboratories. It uses a low power proton beam of 10 MeV to produce neutrons in a beryllium target via nuclear reactions. The neutrons are moderated in a compact system of thermal and cold moderators before they are extracted and transported to the instruments. We will present the MCNP simulations used for the optimisation of neutron flux and brilliance in the extraction channels. In addition, we will introduce a measurement of the neutron yield produced by protons and deuterons and show a comparison with the simulated results to provide a quantitative legitimisation of the applicability of our simulations.

[1] U. Rücker et al.; The Jülich high-brilliance neutron source project; The European Physical Journal Plus, 131; 2016

[2] E. Mauerhofer et al.; Conceptual Design Report, NOVA ERA (Neutrons Obtained Via Accelerator for Education and Research Activities), A Jülich High Brilliance Neutron Source project; 2017

## Investigation of structure and dynamics of liquid surfaces by X-ray pump – optical probe measurements

ID 371

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Structural and dynamical properties of surfaces and interfaces are of pivotal importance for the elucidation of surface phenomena. For example, chemical reactions at surfaces and interfaces are influenced by these properties. Here, we perform optical pump – X-ray probe measurements from liquid-air interface using LISA diffractometer [1], for the first time, installed at P08 beamline at the PETRA III synchrotron radiation source in Hamburg. The diffractometer is specialized for hard X-ray scattering studies of liquid surfaces and interfaces with highly focused X-ray beam. The setup takes advantage of the specific design of LISA, where all scattering studies can be performed without moving the sample. Here, we also describe the extension of this instrument's capabilities by an optical pump - X-ray probe option, which will allow investigations of ultrafast processes at liquid interfaces. This includes installation of a synchronized femtosecond-laser system and optics for directing the laser pulse onto the sample surface. First results from pump-probe X-ray reflectivity experiments at the liquid - air interface has confirmed a time resolution better than 100ps. These new capabilities provide access to structural changes induced via optical excitation, allowing us to understand the non-equilibrium processes on liquid interfaces such as water and mercury. First obtained results on these systems, specifically, from liquid mercury-air interface and air-water interface will be presented here.

[1] B. M. Murphy, B Runge and O. M. Magnussen et al., J. Synchrotron Rad. 21, 45 (2014)

## 2D Ptychography from single semiconductor nanowires

ID 376

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2D dimensional ptychography in Bragg geometry is powerful synchrotron radiation technique to study the shape and strain of the nano-objects. We applied 2D ptychography to determine the axial displacement field of single core-shell-semiconductor nanowires (NWs). The NWs were grown on silicon (111) substrates and are radially composed of a 140 nm GaAs core, 10 nm In<sub>0.10</sub>Ga<sub>0.90</sub>As shell and a 30 nm GaAs outer shell. The NWs are typically 2-3  $\mu\text{m}$  long. 2D ptychography experiment has been performed at beamline ID01 of the ESRF, Grenoble, using coherent x-rays with energy of 9 keV and a beam size of 150 x 200 nm<sup>2</sup> (FWHM). Using a two dimensional detector diffraction patterns were collected at the GaAs (111) Bragg peak. Parallel to the ptychography experiment, the same NWs have been measured using coherent X-ray diffraction imaging (CXDI) to record 3D reciprocal space maps at three different positions along the NW growth axis in a similar way as shown in Ref. [2]. The displacement field was retrieved from 2D ptychography data using the PyNX software [1]. The displacement field along the NW growth axis of NW1 is rather homogeneous, in contrast it appears inhomogeneous for NW2. The CXDI results are in good agreement with the 2D ptychography reconstructions because it shows homogeneous phase at NW1 but phase changes along the growth axis at NW2 are found.

[1] Mandula et. al. J. Appl. Cryst. 49 (2016), 1842-1848

[2] Davtyan. et. al. J. Appl. Cryst. (2017). 50, 673-680

## Focusing fourth generation X-rays: Commissioning results of the NanoMax KB @ MAX IV

ID 377

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*ROBISCH, Anna-Lena (Röntgenphysik Göttingen); KALBFLEISCH, Sebastian (MAX IV Synchrotron Lund); SOLTAU, Jakob (Röntgenphysik Göttingen); ECKERMANN, Marina (Röntgenphysik Göttingen); JOHANSSON, Ulf (MAX IV Synchrotron Lund); SALDITT, Tim (Röntgenphysik Göttingen)*

Fourth generation synchrotrons like MAX IV in Lund, Sweden, provide a brilliance gain of about two orders in magnitude. This puts new challenges on the fabrication of optics, sample stages, detectors, and data analysis chains. Here we focus on the focusing properties of the Kirkpatrick- Baez mirror system at the NanoMAX beamline [1], and present recent commissioning results. We compare alignment tolerances from a geometrical model to direct measurements of the caustic obtained by X-ray waveguide scanning both in focus and in defocus planes, providing a rule of thumb on beam broadening and focal plane shift. The geometrical model is enhanced by coherent wave-optical simulations [2]. With its secondary source, the NanoMAX offers the possibility to change the numerical aperture of the optics (entrance slits) and the coherence properties (secondary source slits) independently. We exploit this to measure the degree of lateral coherence for different settings, and compare the experimental data with partially coherent wave-optical simulations [3].

[1] U. Johansson et al: NanoMAX: A hard x-ray nano probe beamline at MAX IV, Proc SPIE 8851 (2013)

[2] Ch. Morawe, M. Osterhoff: Hard X-Ray Focusing with Curved Reflective Multilayers, XROI (2010).

[3] T. Salditt et al, Partially coherent nano-focused x-ray radiation characterized by Talbot interferometry, Opt Express 19 (2011).

**Beamline P10 'Coherence Applications' at PETRA III**

ID 382

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The Coherence Applications Beamline P10 at PETRA III is dedicated to coherent X-ray scattering experiments using X-ray Photon Correlation Spectroscopy (XPCS) and Coherent Diffraction Imaging (CDI) techniques as well as time-resolved studies of complex liquids (Rheo-SAXS). Mostly, the beamline operates in the energy range of 5-17 keV. It consists of two 12m long experimental hutches (EH1 & EH2) which house various experimental setups. Here, we want to focus on the ultra-small angle X-ray scattering (USAXS) setup at P10 – while the sample position is situated in the first experimental hutch (EH1), the detector is positioned at the end of the second hutch (EH2), which results in a sample to detector distance of around 21.3 m. This long pathway allows it to use a large fraction of the horizontal coherent flux in an unfocused X-ray beam, while providing a fairly strong speckle visibility at 8 keV using state-of-the-art hybrid pixel array detectors. The setup is therefore ideally suited for radiation sensitive samples such as most complex liquids, as the flux per sample area can be considerably reduced using a comparatively large X-ray beam.



## Temperature Sample Environment for the High Field Magnet at the Helmholtz-Zentrum Berlin

ID 390

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The Helmholtz-Zentrum Berlin with its expertise on high magnetic fields for neutron scattering has successfully installed a high field magnet (HFM) that has set a new record for static magnetic fields for neutron scattering. A magnetic field of 26.286 T was reached on October, 16th 2014. Since early 2015 the HFM is integrated in the neutron instrument ExED (TOF) representing a worldwide unique research instrument. For the HFM there are different temperature inserts available: a  $^3\text{He}$ -Insert for temperatures down below 0.8 K, a  $^4\text{He}$  cryostat with a base temperature just below 2 K and a  $^3\text{He}/^4\text{He}$  dilution cryostat. The  $^4\text{He}$  cryostat is equipped with a cold rotation stage powered by a piezo motor. In this presentation we will report about the present status of the temperature sample environment for the HFM, highlight the technical challenges and give an overview of the future developments.

## Interfacial spin-coupling in magnetic bilayers and their role for ultrafast magnetization dynamics

ID 391

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Rare-earth metals exhibit the highest magnetic moments among all atomic elements and show delicate magnetic ordering and concomitant complex photo-induced dynamics. We studied the fluence-dependent magnetization dynamics in Gadolinium (Gd), Terbium (Tb), and in bilayer systems combining both metals, grown on a W(110) substrate. For our investigations the FemtoSpex slicing facility at the synchrotron source BESSY II is ideal since we can probe the magnetization dynamics with X-ray magnetic circular dichroism element-specifically and with a time resolution of 130 fs. All samples show a two-step demagnetization as observed in previous experiments on Gd and Tb [1-4]. Interestingly the static magnetic properties as well as the laser-induced ultrafast spin dynamics of a Gd thin film can be dramatically altered by depositing a few monolayers of Tb on top. Our results further indicate that the interfacial coupling in the bilayer system and the sub-picosecond spin dynamics of the composite system depend on the sample temperature and varies with distance from the interface. Our future studies will profit from the new BMBF-financed DynaMax endstations at the slicing beamline.

[1] M. Wietstruk et al., Phys. Rev. Lett. 106, 127401 (2011).

[2] M. Sultan et al., Phys. Rev. B 85, 184407 (2012).

[3] A. Eschenlohr et al., Phys. Rev. B 89, 214423 (2014).

[4] K. Bobowski et al., J. Phys.: Condens. Matter 29, 234003 (2017).

## Vacuum-compatible hybrid photon counting pixel detector for WAXS, XRD and XRR in the tender X-ray range

ID 392

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A vacuum-compatible hybrid pixel detector has been installed in the UHV reflectometer of the four-crystal monochromator beamline of the Physikalisch-Technische Bundesanstalt (PTB) [1] at the electron storage ring BESSY II in Berlin, Germany. It was developed in cooperation with Dectris and is based on the PILATUS3 100k module. The detector can be used in the entire photon range of the beamline from 10 keV down to 1.75 keV. In comparison to the PILATUS 1M detector used for small-angle scattering (SAXS) and grazing incidence SAXS (GISAXS) [2] it is possible to access larger scattering angles. The water-cooled module can be positioned from  $-55^\circ$  to  $+55^\circ$  with respect to the incoming beam at a distance of about 200 mm from the sample. The quantum efficiency, homogeneity and linearity of the new detector have been investigated. First results of the performance in wide-angle X-ray scattering (WAXS), X-ray diffraction (XRD) and X-ray reflectometry (XRR) will be presented.

[1] M. Krumrey and G. Ulm, Nucl. Instr. and Meth. A 467-468, 1175 (2001)

[2] J. Wernecke, C. Gollwitzer, P. Müller and M. Krumrey, J. Synchrotron Rad. 21, 529 (2014)

## Study of the crystalline structure and oxidation states of the CO and PR of the serie La<sub>0.5-x</sub>Pr<sub>x</sub>Ba<sub>0.5</sub>CoO<sub>3</sub>- with synchrotron radiation

ID 400

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Solid oxide fuel cells (SOFCs) are promising devices to generate clean energy efficiently. Numerous studies are currently underway to reduce the operating temperature of these cells from 800 - 1000°C to temperatures below 700°C (IT-SOFC). Perovskite type materials (ABO<sub>3</sub>) have generated great interest due to their excellent electronic conduction properties and good conduction by oxygen anions at high temperature. This mixed conduction makes them great candidates to be applied as an electrode in IT-SOFCs. In this work, the crystalline structure and the chemical properties of Co and Pr in the series La<sub>0.5-x</sub>Pr<sub>x</sub>Ba<sub>0.5</sub>CoO<sub>3</sub>- ( $0 \leq x \leq 0.5$ ) were studied by X-ray diffraction (XRD) and X-ray Absorption Spectroscopy (XAS) technique, in controlled conditions of atmosphere and temperature. The XRD and XAS data analyzes indicate that these samples present a mixture of cubic and tetragonal phases and at room temperature the cobalt is present in oxidation states +3 and +4 while at higher temperatures it is reduced, mostly presenting an oxidation state +2 and to a lesser extent +3 and +4. It could also be concluded that the oxidation state of Co is very sensitive to temperature and partial pressure of oxygen (pO<sub>2</sub>). On the other hand, we observe that Pr remains almost invariant due to changes in temperature and pO<sub>2</sub> in the samples analyzed. All this suggests that the ease of the Co to change its oxidation state plays a major role in the electrocatalytic activity of the cathode.

## Photothermal radiometry study of heavy ion beam induced modification of thermal properties of graphite

ID 402

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*PROSVETOV, Alexey* (TU Darmstadt); *HAMAOU, Georges* (Multiscale Thermophysics Lab., GRESPI, Université de Reims Champagne Ardenne URCA); *CHIRTOC, Mihai* (Department of Materials and Earth Sciences, Darmstadt Technical University); *HORNY, Nicolas* (Department of Materials and Earth Sciences, Darmstadt Technical University)

The evolution of thermal properties of samples irradiated with GeV Au and U ions at the UNILAC accelerator at GSI was studied using the photothermal radiometry (PTR) technique. PTR measurements allow a depth resolved measurement of the thermal properties of the irradiated samples. In the present study, this technique is applied to characterize the 50-70  $\mu\text{m}$  thick damaged layer on swift heavy ions irradiated graphite samples. Graphite is used in high power accelerator applications as a materials for beam dumps and production targets. Irradiation-induced degradation of its thermal properties leads to a decreased efficiency of dissipation of the heat deposited by the high intensity ion beam and to premature failure. The thickness of the damaged layer calculated by SRIM was experimentally confirmed by Raman spectroscopy and SEM imaging on the sample's cross-section. The results show a significant degradation of thermal effusivity down to 20% of the pristine value and a slight decrease of volumetric heat capacity of irradiated graphite at the maximum reached ion fluence of  $5 \times 10^{13}$   $\text{i}/\text{cm}^2$ . The measured thermal properties of the irradiated layers reflect values characteristic to glassy carbon. The offline studies indicate that the non-contact PTR technique can be applied for in situ studies of beam-induced thermal properties degradation.

## RESEDA: Resonance Spin Echo Spectrometer

ID 404

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RESEDA (Resonance Spin Echo for Diverse Applications) is a high-resolution resonance spin echo spectrometer, operated by the Technische Universität München. It is situated at the end of neutron guide NL5-S at the cold source of the FRM-II research reactor in Garching Germany [1]. In contrast to classical neutron spin echo spectrometers, RESEDA realizes the spin precession by resonance coils rather than voluminous solenoids and thus allows besides spin echo spectroscopy also the use of the MIEZE (Modulation of Intensity by Zero Effort) mode [2]. This technique allows the investigation of quasi elastic scattering of depolarizing samples (i.e. strong incoherent scattering) or under depolarizing sample environments like magnets. Both options give access to a large time and scattering vector range for quasi-elastic measurements. In a recent upgrade program, founded by the BMBF (BMBF-Projekt 05K16WO6: "Longitudinale Resonante Neutronen Spin-Echo Spektroskopie mit Extremem Energie Auflösung"), several components were renewed for a more robust operation and optimized to increase the accessible spin-echo times over more than 6 orders of magnitude (currently:  $1 \times 10^{-5} \text{ ns} < \tau < 5 \times 10^1 \text{ ns}$ ). The poster will present latest developments at RESEDA, few technical specifications and exemplary results for the determination of the diffusion constants in liquid bulk systems.



## In situ time-resolved study of nanoscale diffusion phenomena within Cu/W nano-multilayers using GISAXS

ID 410

**ROMANO BRANDT, Leon Manuel (University of Oxford)**

*SALVATI, Enrico (University of Oxford); PAPADAKI, Chrysanthi (University of Oxford); Prof. KORSUNSKY, Alexander (University of Oxford)*

Nanometric Cu/W multilayers deposited onto Si substrates by RF ion beam sputtering experience a complex residual stress state that arises during deposition and depends on a range of parameters. In addition, the high density of bi-material interfaces in multi-layered systems along with the high volume fraction of grain boundaries within the nanocrystalline layers lead to a large increase in the free energy of the system. These factors promote mass transport, leading to a lower thermal stability and therefore lower reliability of the final multilayer product. In order to understand the diffusion phenomena that begin to occur at unexpectedly low temperatures, Grazing Incidence Small Angle X-Ray Scattering (GISAXS) was used for a temperature and time-resolved study. Additionally, further synchrotron techniques were used for sample characterisation, such as X-Ray Reflectometry (XRR) and Wide-Angle X-Ray Scattering (WAXS). The results will be presented in this talk.

## High-Pressure Hydrogen Cell for in-situ Characterisation of Hydrogen Storage Materials

ID 418

**ASLAN, Neslihan (TUM)**

*PRANZAS, Klaus (HZG); HORSTMANN, Christian (HZG); KOTLYAR, Oleg (HZG); METZ, Oliver (HZG); MÜLLER, Martin (HZG); PISTIDDA, Claudio (HZG); DORNHEIM, Martin (HZG); BUSCH, Sebastian (HZG); LOHSTROH, Wiebke (TUM)*

One way to store hydrogen is the use of Reactive Hydride Composites (RHCs), like  $\text{Ca}(\text{BH}_4)_2\text{-Mg}_2\text{NiH}_4$ . These materials have high capacities and suitable kinetics for hydrogen storage. On the other hand, the challenges of these compounds are the high pressures and temperatures, needed for hydrogen absorption and desorption.  $\text{Ca}(\text{BH}_4)_2\text{-Mg}_2\text{NiH}_4$ , e.g., requires 400 bar  $\text{H}_2$  and  $450^\circ\text{C}$ . Tanks used in industrial applications can even bear 700 bar  $\text{H}_2$  pressure. For the characterisation of such hydrogen storage systems, a high-pressure cell for in-situ neutron studies at pressures up to 700 bar and temperatures up to  $500^\circ\text{C}$  was developed, constructed and tested. The setup of cell and loading station was designed to match the requirements of the different neutron methods and instruments at European neutron facilities. Using this cell for neutron diffraction, small-angle neutron scattering and inelastic neutron scattering experiments, the in-situ characterisation of phase transformations, nanostructures, dynamic properties and hydrogen diffusion in hydrogen storage materials are possible during hydrogen desorption/ absorption. The basic design of the cell includes a heated sapphire capillary. In October 2017 test measurements were performed at different MLZ instruments. The measurements delivered important information for optimisation and further development of setup and corresponding safety procedure.

## Single Nanowire Studies at the GINIX Nanofocus

ID 420

**OSTERHOFF, Markus (Röntgenphysik Göttingen)**

*WALLENTIN, Jesper (Lund University); SOLTAU, Jakob (Röntgenphysik Göttingen); SPRUNG, Michael (DESY); SALDITT, Tim (Röntgenphysik Göttingen)*

Semiconductor nanowires (NWs) are crystalline nanostructures which are intensely researched in areas such as electronics, light-emitting devices, and quantum optics. In particular, nanowire-based solar cells have shown strong development in recent years [1,2]. While the mean crystal parameters can be measured easily for large numbers of NWs, the strain/stress and bending of individual NWs have only by measured at synchrotron radiation facilities. The small length scales of NWs, with a diameter of around 200 nm and a length of about 2  $\mu\text{m}$ , asks for new kinds of focusing optics. We have used Multilayer Zone Plates (MZPs) that can focus hard x-ray energies down to 2D spot sizes below 10 nm [3,4], and hence are suited to spatially map out the NW crystal lattice or act as local stimulus in X-ray beam induced current (XBIC) experiments [5]. We report on recent imaging experiments and in-operando studies on individual shell-core nanowires, which are paralleled by advances on MZP optics and imaging. Real-time data analysis resolves another bottleneck we faced at recent beamtimes.

[1] J. Wallentin et al: InP Nanowire Array Solar Cells Achieving 13.8% Efficiency by Exceeding the Ray Optics Limit, *Science* 339 (2013).

[2] M. Borgström et al: Nanowires With Promise for Photovoltaics, *IEEE JSTQE* 17 (2011).

[3] F. Döring et al: Sub-5 nm hard x-ray point focusing by a combined Kirkpatrick-Baez mirror and multilayer zone plate, *Opt. Express* 21 (2013).

[4] M. Osterhoff et al: Towards multi-order hard X-ray imaging with multilayer zone plates, *J. Appl. Cryst.* 46 (2015).

[5] J. Wallentin et al: Hard X-ray Detection Using a Single 100 nm Diameter Nanowire, *Nano Letters* 14 (2014).

## Fragment Screening on Protein Kinase A and PIM1-Kinase

ID 423

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Fragment screening has been developed into a routine method in the drug discovery process. Compared to high-throughput screening, fragment libraries can be much smaller in size and are therefore amenable to crystallographic screening, either directly or after a suitable prescreening method. Particularly, synchrotron beamlines, such as MX14-2 at Helmholtz-Zentrum Berlin, dedicated to fragment screening allow data collection of numerous data sets within a short time period.[1] We used a 361 compound library developed in our laboratory [2,3] to screen 2 protein kinases - protein kinase A (PKA) and PIM1. As a prescreening method we used a thermal shift assay (TSA). Here, 31 fragments were detected for PKA and 52 for PIM1. These subsets were then selected for crystallographic screening where we obtained 15 complex structures for PKA and an additional 13 for PIM1, revealing a high crystallographic hit rate. Observed hits in the TSA assay are deviating for both kinases and only a single fragment was crystallographically observed binding to both kinases. Results from the TSA and crystallographic screening, together with observed binding motifs for both kinases will be presented.

[1] Mueller, U. et al. (2012) J. Synchrotron Radiat. 19, 442-449.

[2] Köster, H. et al. (2011) J. Med. Chem. 54, 7784-7796.

[3] Schiebel, J. et al. (2016) ACS Chem. Biol. 11, 1693-1701.



## Pulsed Laser Deposition setup for in-situ experiments with at neutron and synchrotron sources

ID 436

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Phenomena and effects on interfaces and in individual atomic layers play a key role in the modern material science, especially in the field of nanotechnologies (nanoelectronics, photonics, plasmonics, etc). The pulsed laser deposition (PLD) technique is a versatile tool with a capability of using a wide variety of materials to produce such structures. Neutron radiation offers a unique possibility for in-situ investigation of multilayer thin films growing processes and for investigation of the asgrown structures. In particular, the method of spin-sensitive neutron polarization reflectometry (NPR) (in the low temperatures ( $<15$  K) and high magnetic fields ( $>1$  T)) is extremely sensitive for the structural and magnetic properties of materials. Moreover, synchrotron sources offer a unique tool for in-situ characterization of the initial growth stages and interfaces of thin films and multilayers. XPS, XRD, XMCD, SMS - this is not a complete list of great techniques for in-situ research. Drawing on our own in situ PLD experience at synchrotrons and neutron facilities and in view of the very wide range of scientific tasks that can be solved by such approach, we have developed a series of mobile PLD setups for various in-situ and in-vacuo synchrotron and neutron techniques. All devices have a mobile configuration and have to be compatible with the existing and future beamlines and instruments. In the presentation, we will give an overview of the experimental chambers and concepts and their application fields. Also, some results of our in-situ PLD investigations with at PETRA III will be presented.



## Structure and Dynamics of Confined Liquids Studied by an X-ray Surface Force Apparatus

ID 442

**MEZGER, Markus (Johannes Gutenberg University Mainz, Institute of Physics)**

*MEROLA, Claudia (TU Wien, Institute for Applied Physics); WEISS, Henning (Max Planck Institut for Polymer Research, Mainz); CHENG, Hsiu-Wei (TU Wien, Institute for Applied Physics); MARS, Julian (Max Planck Institut for Polymer Research, Mainz); VALTINER, Markus (TU Wien, Institute for Applied Physics); HONKIMÄKI, Veijo (ESRF-The European Synchrotron, Grenoble)*

Structure and dynamics of confined liquids were probed by an in-situ X-ray surface force apparatus in plane-cylinder geometry. Our novel device can shear, compress and decompress soft matter within a precisely controlled slit pore confinement. Complementary structural information is obtained by X-ray scattering and simultaneous force measurements. Here, we present results on the structural relaxation of confined liquid crystals (LCs) as reaction to external stimuli. Experiments using high energy x-rays were performed at ID31, ESRF. For the smectic LC 8CB we investigated defect-formation and relaxation processes during subsequent compression and decompression cycles. In the second example, we investigate a thin film of the wet ionic liquid C10mim+Cl<sup>-</sup> in the columnar LC phase [1]. The observed mesoscopic orientation induced by oscillatory shear is explained by the anisotropic mobility of the amphiphilic cations.

[1] H.-W. Cheng et al., Langmuir 34, 2637 (2018)



## Tuesday, Sept. 18<sup>th</sup>, 2018

Each field is linked to the session's start page. Just click on the title!

<b>09:00</b>	Welcome by Projekträger DESY			<b>2001</b>
<b>09:15</b>	<b>Plenary Talk</b> Extraterrestrial dust on earth – Micrometeorites and traces of nearby supernovae <i>J. Feige</i>			<b>2001</b>
<b>10:00</b>	<b>Keynote</b> Magnetic excitations in the quadrupolar ordered CeB <sub>6</sub> <i>D. Inosov</i>			<b>2001</b>
<b>10:30</b>	Coffee break			
<b>11:00</b>	<b>PS 3</b> Life sciences  <b>2001</b>	<b>PS 4</b> Magnetism and quantum phenomena  <b>0001</b>	<b>MS 3</b> Novel developments in time resolved techniques  <b>1801</b>	<b>MS 4</b> Innovation and industry  <b>0350</b>
<b>12:30</b>	Lunch break			
<b>14:00</b>	<b>PS 1</b> Instrumentation and methods  <b>2001</b>	<b>PS 8</b> Functional materials and materials science  <b>0001</b>	<b>MS 3</b> Novel developments in time resolved techniques  <b>1801</b>	<b>MS 5</b> Digital agenda  <b>0350</b>
<b>15:30</b>	Coffee break			
<b>16:00</b> - <b>17:30</b>	<b>Poster session 2</b>			



## Extraterrestrial dust on Earth - Micrometeorites and traces of nearby Supernovae

Tuesday, 09:15  
MW2001  
ID 409

*FEIGE, Jenny (TU Berlin)*

As our Earth moves through space, it traps particles originating from within our Solar System and from the surrounding interstellar medium. Each year, about 30.000 tons of interplanetary dust accumulate on Earth. A large fraction can be found as micrometeorites on the Earth's surface - submillimetre-sized particles liberated from larger bodies such as asteroids and comets. Additionally, interstellar dust signatures from supernovae were observed in deep-sea archives, indicating recent nearby stellar explosions. We analysed sediment samples from a depth of 4200 m below the sea surface originating from the Indian Ocean for their  $^{60}\text{Fe}$  content. This long-lived radionuclide, with a half-life of 2.6 million years, is produced and ejected by exploding stars. Using accelerator mass spectrometry (AMS) at ANU, Canberra, we detected an excess of  $^{60}\text{Fe}$  corresponding to a time period of 2-3 million years ago. Dating of the samples was achieved with AMS measurements (HZDR, Dresden, and VERA, Vienna) of cosmogenic  $^{10}\text{Be}$  and  $^{26}\text{Al}$  - both are long-lived radionuclides produced in the Earth's atmosphere. Micrometeorites contribute to a continuous extraterrestrial influx of  $^{10}\text{Be}$ ,  $^{26}\text{Al}$ , and possibly  $^{60}\text{Fe}$ . These cosmogenic radionuclides accumulate within the microparticles as they are irradiated by cosmic rays in space. Measuring the concentrations of  $^{10}\text{Be}$ ,  $^{26}\text{Al}$ , and  $^{60}\text{Fe}$  within a single micrometeorite yields information on its cosmic ray exposure duration and its origin within our Solar System.



## Magnetic excitations in the quadrupolar ordered CeB<sub>6</sub>

Tuesday, 10:00  
MW2001  
ID 280

*INOSOV, Dmytro (TU Dresden)*

Cerium hexaboride is a textbook example of an f-electron system hosting an exotic antiferroquadrupolar order. Even after decades of intense studies, it still lacks a complete theoretical description, and experiments continue to present puzzles in the form of unexpected observations. In recent years, we have scrutinized the magnetic excitation spectrum of CeB<sub>6</sub> and its doped derivatives such as Ce<sub>1-x</sub>La<sub>x</sub>B<sub>6</sub> and Ce<sub>1-x</sub>Nd<sub>x</sub>B<sub>6</sub>. In this keynote lecture, I would like to present our most recent results with a focus on magnetic-field and doping dependence of spin correlations and collective magnon excitations in these systems. We find clear signatures of long-range RKKY interactions between the f-electron multipoles in the diffuse quasielastic response in zero magnetic field that sensitively depends on charge doping. Further, field-induced collective magnon modes have been mapped out in fields up to 14.5 T applied along different crystallographic directions, revealing new high-field magnetic excitations that were not previously detected in electron spin resonance (ESR) or any other probes. These modes exhibit a significant anisotropy with respect to the field direction, offering a new stepping stone for the theoretical understanding of multipolar excitations in the antiferroquadrupolar phase of CeB<sub>6</sub>.



**Tuesday – September 18<sup>th</sup>, 2018**  
**11:00-12:30**

## **Parallel session 3**

### **Live sciences**

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:00-11:15**

*Felix Roosen-Runge*

Towards crowding in the eye lens: dynamics in aqueous solutions of crystallin proteins

**11:15-11:30**

*Tanja Schulz-Mirbach*

The rolling stones live: Hard X-ray phase contrast and neutron imaging allow for the in-situ visualization of otolith motion and associated structures in the fish ear

**11:30-11:45**

*Andreas Stadler*

Dynamics of denatured and native bovine serum albumin: A neutron spectroscopy study

**11:45-12:00**

*Dennis M. Noll*

In Situ Stopped-Flow SANS Study of the Growth of Poorly Water-Soluble Drug Nanoparticles Prepared by Antisolvent Precipitation

**12:00-12:15**

*Saskia Bannister*

Elucidation of light-induced structural changes of aureochrome and its recovery kinetics by small-angle X-ray scattering

**12:15-12:30**

*Volker Schünemann*

Low frequency modes in iron proteins explored with the <sup>57</sup>Fe nuclear probe

**Towards crowding in the eye lens: dynamics in aqueous solutions of crystallin proteins****Tuesday, 11:00  
MW 2001  
ID 55****ROOSEN-RUNGE, Felix (Lund University)***MYUNG, Jin Suk (Lund University); SCHURTENBERGER, Peter (Lund University); STRADNER, Anna (Lund University, Division of Physical Chemistry)*

The function of the eye is dependent on a transparent, refractive, and deformable eye lens. These physical properties are realized by a crowded mixture of mainly crystallin proteins within the eye lens cells. The underlying biophysical mechanisms are not only of fundamental interest, but highly relevant to better understand and treat eye conditions such as presbyopia and cataract. We present experimental and simulation data on nanosecond dynamics in solutions of  $\alpha$ ,  $\beta$  and  $\gamma$  crystallins as model systems for the cytoplasm in the eye lens. While cage diffusion and gradient diffusion in  $\alpha$  crystallin solutions are consistent with hard sphere systems [1,2], solutions of  $\gamma$  crystallins show short-range attraction, resulting in a significantly reduced cage diffusion [1], and critical slowing down of the gradient diffusion [3].  $\beta$  crystallins appear to have only weak attractive interactions, causing smaller effects than for  $\gamma$  crystallin. Based on the dynamics characterised in mono-component solutions, we discuss the effects of mutual protein interaction in mixed solutions of crystallins on the dynamics and arrest behavior. For both mixtures of  $\alpha / \gamma$  crystallins and  $\beta / \gamma$  crystallins, non-additive effects of the diffusion are observed, suggesting mutual interaction between the crystallins [4].

[1] S Bucciarelli et al. Sci.Adv. 2 (2016) e1601432

[2] G Foffi et al. PNAS 111 (2014) 16748

[3] S Bucciarelli et al. JPCL 6 (2015) 4470

[4] A Stradner et al. PRL 99 (2007) 198103

## The rolling stones live: Hard X-ray phase contrast and neutron imaging allow for the in-situ visualization of otolith motion and associated structures in the fish ear

Tuesday, 11:15

MW2001

ID 235

**SCHULZ-MIRBACH, Tanja (LMU, Department Biology II)**

*OLBINADO, Margie P. (ESRF, Beamline ID19); MITTONE, Alberto (ESRF, Beamline ID17); SCHILLINGER, Burkhard (TUM-FRM II, MLZ-ANTARES); LADICH, Friedrich (University of Vienna, Dept. of Behavioural Biology); MELZER, Roland R. (Bavarian State Collection of Zoology (ZSM)); HEß, Martin (LMU Department Biology II)*

In the fish ear, the basics of the relative motion between the calcareous otolith and the underlying sensory epithelium (SE) are still elusive. The few experimental studies and mathematical modeling indicated that otolith motion is mass-, shape- and frequency-dependent. Yet, it is widely unknown how 3D-otolith shape affects otolith motion and if this translates into differential patterns of ear stimulation. At the ESRF, we successfully designed a set-up to visualize otolith motion in-situ using hard X-ray phase contrast imaging and tested the effects of otolith shape and mass and different swimbladder types. The studied species thus differed in otolith and swimbladder morphology. *Steatocranus tinanti* has fusiform otoliths and a vestigial swimbladder, *Etroplus maculatus* displays rhomboid otoliths and possesses a swimbladder contacting the ears. In goldfish (*Carassius auratus*), the otoliths are needle-shaped and a chain of ossicles and ligaments transmits vibrations from the swimbladder to the ears. In a water-filled tank, pure tones of 0.1 or 0.2 kHz were presented to a single otolith embedded in agarose, to a fish head, and to a whole fish. Additionally, we investigated the native structure of the otolithic membrane that mediates the motion between otolith and SE using neutron tomography and neutron grating interferometry imaging. We will present outcomes of our combined study using synchrotron and neutron imaging which aims to elucidate otolith function in the fish ear.

## Dynamics of denatured and native bovine serum albumin: A neutron spectroscopy study

Tuesday, 11:30  
MW2001  
ID 112

**STADLER, Andreas (FZ Jülich)**

*AMESEDER, Felix (FZ-Juelich, JCNS-1); RADULESCU, Aurel (Jülich Centre for Neutron Science - Outstation at MLZ); KHANEFT, Marina (JCNS at MLZ); LOHSTROH, Wiebke; HOLDERER, Olaf; Dr FALUS, Peter (Institut Laue-Langevin); RICHTER, Dieter (Forschungszentrum Jülich JCNS)*

A characteristic property of unfolded and disordered proteins is their high molecular flexibility, which enables the exploration of a large conformational space. We present neutron scattering experiments on the dynamics of denatured and native folded bovine serum albumin (BSA) in solution over the full time-range that is accessible via neutron spectroscopy (1,2). In a first set of experiments protein dynamics of unfolded and folded BSA were investigated on the ps to ns timescale using neutron time-of-flight and backscattering spectroscopy (1). A significant dynamical heterogeneity in the native folded protein was observed. Chemical denaturation has a drastic effect on the ps to ns motions of the protein. Anomalous diffusion in denatured BSA was found to show essentially characteristic properties of heterogeneous dynamics, caused by a distribution of exponential diffusive processes. Using neutron spin-echo (NSE) experiments, we observed a high internal flexibility of denatured BSA (2). Internal motions measured by NSE were described using concepts based on polymer theory. The contribution of residue-solvent friction was accounted for using the Zimm model including internal friction. Disulphide bonds forming loops of amino acids of the peptide backbone have a major impact on internal dynamics of denatured BSA that can be interpreted with a reduced set of Zimm modes

1. Ameseder et al. Phys Chem Chem Phys, 20, 5128-5139
2. Ameseder et al. J. Phys. Chem. Lett, 9, 2469-2473



## In Situ Stopped-Flow SANS Study of the Growth of Poorly Water-Soluble Drug Nanoparticles Prepared by Antisolvent Precipitation

Tuesday, 11:45  
MW2001  
ID 216

*NOLL, Dennis M. (Institute for Crystallography and Structural Physics, FAU Erlangen- Nürnberg)*

*SCHULDES, Isabel (Institute for Crystallography and Structural Physics, FAU Erlangen- Nürnberg); ZECH, Tobias (Institute for Crystallography and Structural Physics, FAU Erlangen- Nürnberg); UNRUH, Tobias (FAU Erlangen- Nürnberg)*

Using the stopped-flow technique for classical in situ small-angle neutron scattering (SANS) experiments enables to study fast structure formation processes like the formation and growth of organic nanoparticles (NPs) with a temporal resolution in the regime of milliseconds. The statistics of short time frames can be enhanced during the post processing by adding the detected neutrons of each frame for a high amount of repetitive mixing processes of the stopped-flow technique. First in situ experiments at the KWS-2 instrument (MLZ, Garching) show the reliability of the combination of the stopped-flow technique with SANS. The early growth of poorly-water soluble active pharmaceutical ingredients (APIs) NPs during the antisolvent precipitation process has been studied. The SANS data has been acquired with a high temporal resolution of 2 ms during the first 2 s and by performing up to 400 mixing processes for each sample system. To further enhance the statistics for the analysis, the frames have been binned to a final temporal resolution of 20 ms. The influence of three structurally different and commonly used surfactants on the growth kinetics and the ripening of the model API fenofibrate has been studied and first analyses reveal an early beginning of ripening already after 250 ms for all surfactant systems and increased growth kinetics combined with inhibited ripening for fenofibrate NPs solubilized by micelles.



## Elucidation of light-induced structural changes of aureochrome and its recovery kinetics by small-angle X-ray scattering

Tuesday, 12:00

MW2001

ID 116

**BANNISTER, Saskia (Bielefeld University, PCIII)**

*HERMAN, Elena (Bielefeld University, PCIII); HELLWEG, Thomas (Bielefeld University, PCIII); KOTTKE, Tilman (Bielefeld University, PCIII)*

Aureochromes function as blue-light-regulated transcription factors in algae. Their basic region leucine zipper (bZIP) effector domain binds DNA specifically while a light-, oxygen-, or voltagesensitive (LOV) domain acts as the sensor. Due to the inversed arrangement of sensor and effector, aureochromes are interesting for studying their mechanism and for the engineering of new optogenetic tools. By applying small-angle X-ray scattering (SAXS) we pursue two main targets, namely the elucidation of light-induced structural changes of the receptor in solution and the analysis of the recovery kinetics from its light state back to its dark state. However, SAXS on photoreceptors is challenging. First, dark conditions need to be absolutely strict to avoid conversion of the highly sensitive receptor. Second, the analysis under illumination needs to ensure full conversion. Therefore we have established SAXS experiments under rigorous control of light. Here, we reveal light-induced structural changes of the photoreceptor and its recovery kinetics.

Banerjee, A., Herman, E., Serif, M., Maestre-Reyna, M., Hepp, S., Pokorny, R., Kroth, P. G., Essen, L.-O., Kottke, T. (2016), *Nucleic Acids Res.* 44(12), 5957-5970.

## Low frequency modes in iron proteins explored with the $^{57}\text{Fe}$ nuclear probe

Tuesday, 12:15  
MW2001  
ID 366

**SCHÜNEMANN, Volker** (Technische Universität Kaiserslautern)

Synchrotron based nuclear resonance scattering techniques are ideal tools to investigate electronic and dynamic properties of iron centers in chemical and biological systems. During the last years Nuclear Inelastic Scattering (NIS) also called Nuclear Resonance Vibrational Spectroscopy (NRVS) has been used to detect iron based vibrational modes of iron proteins and chemical model complexes. We have investigated the vibrational modes of iron-sulphur-proteins [1], both experimentally via NIS and theoretically by density functional theory (DFT) calculations in the spectral region from  $\sim 5$  to  $\sim 800$   $\text{cm}^{-1}$ . DFT calculations coupled with molecular mechanics allow to investigate not only iron ligand interactions but also solvent effects. The effect of solvent on the low frequency modes as well as on the iron ligand modes of the ligand-binding oxidase from the bacterium *Geobacillus kaustophilus* (Gklox) [2] will be presented. By means of a novel cryostat especially designed for NIS experiments down to  $T = 1.9$  K installed at beamline P01, PETRA III, it is now possible to explore very low frequency modes down to the range of a few wavenumbers by avoiding multiphonon excitations. First results with this sample environment on Gklox as well as on a chemical spin crossover (SCO) complex will be presented.

This work was supported by the BMBF via 05K14UK1 and 05K16UKA.

1) I. Faus, A. Reinhard, S. Rackwitz, J. A. Wolny, K. Schlage, H.-C. Wille, A. Chumakov, S. Krasutsky, P. Chaignon, C. D. Poulter, M. Seemann, V. Schünemann, *Angew. Chem. Int. Ed.* 54(43) (2015) 12584.

2) R. Kositzki, S. Mebs, J. Marx, J.J. Griese, N. Schuth, M. Högbom, V. Schünemann, M. Haumann *Inorg. Chem.* 55 (19) (2016) 9869.



**Tuesday – September 18<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Parallel session 4***

# ***Magnetism and quantum phenomena***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:00-11:15**

*Alexander Backs*

Vortex Lattice Domain Formation in the Type-II/1 Superconductor Niobium

**11:15-11:30**

*Natalija van Well*

Magnetic order in the Cs<sub>2</sub>CuCl<sub>4</sub>-xBr<sub>x</sub> mixed system

**11:30-11:45**

*Dirk Honecker*

Magnetic small angle neutron scattering of two-phase bulk ferromagnets

**11:45-12:00**

*Stephan Geprägs*

Static magnetic proximity effects and spin Hall magnetoresistance in Pt/Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>

**12:00-12:15**

*Dieter Lott*

Observation of a chirality created exchange bias effect

**12:15-12:30**

*Hans-Christian Wille*

Probing electronic and magnetic properties in iridates using nuclear resonant scattering on <sup>193</sup>Ir



## Vortex Lattice Domain Formation in the Type-II/1 Superconductor Niobium

Tuesday, 11:00  
MW0001  
ID 277

**BACKS, Alexander (Heinz Maier-Leibnitz Zentrum, Technische Universität München)**

*REIMANN, Tommy (Heinz Maier-Leibnitz Zentrum, Technische Universität München); SCHULZ, Michael (Heinz Maier-Leibnitz Zentrum, Technische Universität München); PIPICH, Vitaliy (Jülich Centre for Neutron Science, Heinz Maier-Leibnitz Zentrum); MUEHLBAUER, Sebastian (Heinz Maier-Leibnitz Zentrum); BÖNI, Peter (Technische Universität München)*

Besides the well-known vortex lattice (VL), type-II/1 superconductors exhibit the intermediate mixed state (IMS), where VL domains and Meissner domains coexist, due to an attractive component of the VL interaction [1]. Despite some work in the 1970s, no detailed theoretical model for the IMS exists. Primary obstacles are the inapplicability of most standard models of superconductivity and the importance of demagnetization effects and vortex pinning. We have readdressed the IMS in bulk niobium by combining several neutron based techniques [2],[3]. Using small angle scattering, we characterized the VL crystallography. For the larger IMS domains, we used ultra-small angle scattering to investigate the domain morphology. Additionally, neutron grating interferometry revealed information about the spatial distribution of IMS domains. Following a field cooling protocol, we find that while the macroscopic magnetic properties remain homogeneous, vortices are gradually rearranged microscopically from a uniform VL to inhomogeneous domains. Surprisingly, this process takes place below the macroscopic freezing transition of the VL. The IMS domains form at a preferred length scale which remains nearly unchanged with temperature. These results shed new light on the detailed formation of the IMS in bulk superconductors.

[1] E. H. Brandt and M. P. Das, J. Supercond. Novel Magn. 24, 57 (2011)

[2] T. Reimann, et al, Nat. Com. 6, 8813 (2015)

[3] T. Reimann, et al, Phys. Rev. B 96(14), 144506 (2017)

## Magnetic order in the $\text{Cs}_2\text{CuCl}_4\text{-xBr}_x$ mixed system

Tuesday, 11:15  
MW0001  
ID 111

**VAN WELL, Natalija** (Laboratory of Crystallography, University of Bayreuth, Bayreuth)

ZAHARKO, Oksana (Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institute); DELLEY, Bernard (Condensed Matter Theory Group, Paul Scherrer Institute); SKOULATOS, Markos (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München); GEORGII, Robert (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München); VAN SMAALEN, Sander (Laboratory of Crystallography, University of Bayreuth); RÜEGG, Christian (Research with Neutrons and Muons)

$\text{Cs}_2\text{CuCl}_4$  and  $\text{Cs}_2\text{CuBr}_4$  are anisotropic triangular lattice materials, where  $\text{Cu}^{2+}$  ions with  $S=1/2$  form frustrated 2D planes [1,2]. The mixed system  $\text{Cs}_2\text{CuCl}_4\text{-xBr}_x$  is used to study the effect of controlled-quenched disorder in the spin superstructure lattice. Preferential occupation of Cl/Br leads to selective disorder in the lattice and some well-ordered structures. The neutron diffraction investigation of the magnetic phase diagram of  $\text{Cs}_2\text{CuCl}_4\text{-xBr}_x$  provides detailed information about the influence of a specific Br concentration on the magnetic structure and help to clarify, how small modifications of the local  $\text{Cu}^{2+}$  environment influence the exchange couplings and frustration in these compounds. Two different long-range ordered magnetic phases are found in this mixed system, and the first overview of the low-temperature magnetic phases of  $\text{Cs}_2\text{CuCl}_4\text{-xBr}_x$  will be presented. The density functional theory (DFT) calculations show values of exchange coupling constants  $J$ ,  $J'$  for some ordered compositions of this mixed system. Furthermore, we start also studies in a magnetic field, because the quantum critical points are exciting, which may be also studied in the presence of disorder and potential spin-liquid phases in this mixed system.

[1] R. Coldea et al. J. Phys.: Condens Matter 1996, 8, 7473-7491

[2] T. Ono et al. J. Phys. Soc. Jpn. 2005, 74, 135



## Magnetic small-angle neutron scattering of two-phase bulk ferromagnets

Tuesday, 11:30  
MW0001  
ID 275

*HONECKER, Dirk (Institut Laue-Langevin)*

*MICHELS, Andreas (University of Luxembourg)*

Small-angle neutron scattering (SANS) is a powerful method to resolve the magnetic microstructure in the bulk and on length scales between about 1 – 100 nm. We have used this technique to study Fe-based two-phase nanocrystalline alloys. In the investigated soft magnetic heterogeneous nanocomposites, the jump of the magnetization at the phase boundary between particles and matrix gives rise to a dipolar stray field, which represents a non-negligible source of spin disorder. With the help of a micromagnetic description of the magnetic SANS cross section, it can be shown that magnetic anisotropy and magnetostatic field in the sample decisively determine the properties of the strongly magnetic field-dependent scattering, such as the asymptotic power-law behaviour, the range of spin-misalignment correlations and angular anisotropies. In particular, the 'cloverleaf'- shaped angular anisotropy, which was observed for several nanostructured magnetic materials, can be clearly attributed to jumps of the magnetization at internal interfaces. Moreover, the micromagnetic approach allows one to extract quantitatively essential features of the spin structure from the field behaviour of the magnetic SANS, for instance, the average exchange interaction as well as the strength of magnetic anisotropy and magnetostatic fields.



## Static magnetic proximity effects and spin Hall magnetoresistance in Pt/Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>

Tuesday, 11:45  
MW0001  
ID 407

**GEPRÄGS, Stephan (Walther-Meissner-Institut, Bayerische Akademie der Wissenschaften)**

*KLEWE, Christoph (Advanced Light Source, Lawrence Berkeley National Laboratory); MEYER, Sibylle (Walther-Meissner-Institut, Bayerische Akademie der Wissenschaften); COLLINS, Stephan P. (Diamond Light Source Ltd, Harwell Sci & Innovat Campus); OLLEFS, Katharina (European Synchrotron Radiation Facility (ESRF)); WILHELM, Fabrice (European Synchrotron Radiation Facility (ESRF)); ROGALEV, Andrei (European Synchrotron Radiation Facility (ESRF)); GOENNENWEIN, Sebastian T. B. (Institut für Festkörper- und Materialphysik, Technische Universität Dresden); OPEL, Matthias (Walther-Meissner-Institut, Bayerische Akademie der Wissenschaften); KUSCHEL, Timo (Center for Spinelectronic Materials and Devices, Department of Physics, Bielefeld University); GROSS, Rudolf (Walther-Meissner-Institut, Bayerische Akademie der Wissenschaften)*

Heavy metal/ferromagnetic insulator (HM/FMI) heterostructures provide a unique platform for the generation and detection of pure spin currents in the fields of spintronics and spin caloritronics. In these HM/FMI heterostructures, the transport of the spin current across the HM/FMI interface is of key importance. Thereby the quality of the interface highly affects the interfacial interaction between the angular momentum and the HM conduction electrons. To investigate possible intermixing effects in HM/FMI heterostructures, we study Pt/Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> bilayers with differently arranged layers and different interface properties using x-ray magnetic circular dichroism and x-ray resonant magnetic reflectivity combined with magnetotransport studies. For standard Pt/Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> bilayer samples with sharp and clean interfaces we do not detect any induced magnetic moments in Pt. These samples show an angle-dependent magnetoresistance behavior, which is consistent with the established spin Hall magnetoresistance [1]. In inverted Y<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub> /Pt bilayer samples with gradual and intermixed interfaces, however, Pt displays a finite induced magnetic moment comparable to that of all-metallic Pt/Fe bilayers. In these samples, we find a superposition of the spin Hall magnetoresistance and the anisotropic magnetoresistance. Both effects can be disentangled from each other due to their characteristic temperature dependence.

[1] H. Nakayama et al., Phys. Rev. Lett. 110, 206601 (2013).

## Observation of a chirality created exchange bias effect

Tuesday, 12:00  
MW0001  
ID 239

*LOTT, Dieter (Helmholtz-Zentrum Geesthacht)*

*PHILIPPI-KOBS, Andre (DESY)*

The exchange bias effect refers to the unidirectional shift of the hysteresis loop of a magnetization curve, conventionally that of a ferromagnet in direct contact to an antiferromagnet. Chiral magnetism, on the other hand, that manifests in the existence of skyrmions or chiral domain walls, offers an alternative way for creating anisotropies in magnetic materials that might have large potential for being used in future spintronic devices. Alloys of rare-earth elements and 3d transition metals (RE-TM) became recently in the focus of attention due their rich variety of magnetic effects owed to the different anisotropies of both material classes [1-4]. Here we show experimental evidence for a new type of in-plane exchange bias effect present at room temperature that is created from a chiral  $90^\circ$  domain wall at the interface of a ferrimagnetic RE-TM (DyCo) layer in contact to a ferromagnetic (NiFe) layer at room temperature. The chiral interfacial domain wall in the here presented system forms due to the presence of macroscopic Dzyaloshinskii-Moriya interaction in the ferrimagnetic layer and its orthogonal exchange coupling to the ferromagnetic layer at the interface. As a consequence of the fixed chirality of the interfacial domain wall, the shift of the hysteresis loop can be reversed by changing the orientation of the out-of-plane magnetization of the ferrimagnetic layer. The chirality created tunable exchange bias is very robust against high in-plane magnetic fields and does not show any aging effects after multiple reversals of the exchange bias direction, therefore, it overcomes the limitations of conventional exchange bias systems. Such chirality based EB systems may be of crucial importance for the development of future applications in the field of magnetic sensors and data storage devices.

[1] S. Mangin et.al, Phys. Rev. B 80, 224424 (2009), S. Mangin et.al, Phys. Rev. Lett. 82, 4336 (1999).

[2] Chen, K., Lott, D. et al., Phys. Rev. B 91, 024409 (2015).

[3] Chen, K., Lott, D. et al., Sci. Rep. 5, 18377 (2015).

[4] F. Radu, R. Abrudan, I. Radu, D. Schmitz, H. Zabel, Nat. Communications 3, 715 (2012).

## Probing electronic and magnetic properties in iridates using nuclear resonant scattering on $^{193}\text{Ir}$

Tuesday, 12:15

MW0001

ID 301

**WILLE, Hans-Christian (Deutsches Elektronen-Synchrotron)**

*ALEXEEV, Pavel (Deutsches Elektronen-Synchrotron); SERGEEV, Ilya (DESY); LEUPOLD, Olaf (Deutsches Elektronen-Synchrotron DESY); MCMORROW, Des (London Centre for Nanotechnology and Department of Physics and Astronomy); PERRY, R.S. (London Centre for Nanotechnology and Department of Physics and Astronomy); HUNTER, E.C. (London Centre for Nanotechnology and Department of Physics and Astronomy); RÖHLSBERGER, Ralf (DESY / University of Hamburg)*

Understanding the physical properties of systems which are simultaneously subject to strong spinorbit coupling (SOC) and electron correlations is an en vogue research area and triggered intensive studies e.g. by neutron and x-ray spectroscopy methods. These studies revealed novel electronic and magnetic phases in 4d and 5d materials. At one level, SOC introduces another competing energy scale, producing unexpected electronic states. This is the case for the so-called spin-orbit Mott insulator in iridate perovskites which would be expected to be metallic in the absence of SOC. At another, more profound level, the SOC fully entangles spin and orbital degrees of freedom such that the magnetic interactions acquire an anisotropic, bond-directional nature - the Kitaev interaction - augmenting the conventional isotropic Heisenberg term in 3d systems. Nuclear Resonant Scattering (NRS) is sensitive to electronic and magnetic properties with an enhanced sensitivity to the direction of magnetic moments compared to other methods. In addition, the case of the 73 keV transition in iridium has the advantage that the penetration depth and the focusing options allows for studies at high pressure induced metal insulator transitions and small sample volumina in general. We present first NRS studies at the  $^{193}\text{Ir}$  transition and studies on iridates with an emphasize on the magnetic structure in the strontium iridate  $\text{Sr}_2\text{IrO}_4$  where an out of plane component of the magnetic moment has been observed. To perform the experiments the development of a new monochromator for 73 keV and an efficient fast multi-element Avalanche Photodiode Detector have been essential [1].

[1] <https://arxiv.org/abs/1804.06708s>



**Tuesday – September 18<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Micro symposium 3***

### ***Novel developments in time resolved techniques***

**MW1801**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>11:00-11:15</b> <i>Mario Sauppe</i> X-Ray Movie Camera: A novel approach for time-resolved single-particle imaging
<b>11:15-11:30</b> <i>Markus Guehr</i> The new URSA-PQ instrument for molecular photoenergy conversion at FLASH
<b>11:30-11:45</b> <i>Robert Moshhammer</i> Atomic and Molecular Physics Experiments with the REMI Endstation at FLASH2
<b>11:45-12:00</b> <i>Till Jahnke</i> Time-resolved studies of small molecules employing coincidence detection techniques
<b>12:00-12:15</b> <i>Benjamin Stadtmüller</i> New insights into ultrafast phenomena of solids by time-resolved spectroscopy with fs-XUV laboratory light sources
<b>12:15-12:30</b> Discussions



## X-Ray Movie Camera: A novel approach for time-resolved single-particle imaging

Tuesday, 11:00  
MW1801  
ID 433

**SAUPPE, Mario (TU Berlin)**

*BARI, Sadia (DESY); BISCHOFF, Tobias (TU Berlin); BOLL, Rebecca (XFEL); BOMME, Cédric (DESY); BOSTEDT, Christoph (Arg. Nat. Lab., Northw. Univ.); DÖRNER, Simon (DESY); DÜSTERER, Stefan (DESY); ERK, Benjamin (DESY); FEIGL, Torsten (optiX fab); FLÜCKIGER, Leonie (TU Berlin, La Trobe Univ.); GORKHOVER, Tais (PULSE, LCLS); HEILRATH, Andrea (TU Berlin); KOLATZKI, Katharina (TU Berlin); KUMAGAI, Yoshiaki (Arg. Nat. Lab.); LANGBEHN, Bruno (TU Berlin); MONSERUD, Nils (MBI); MÜLLER, Erland (DESY); MÜLLER, Jan Philippe (TU Berlin); PASSOW, Christopher (DESY); RAMM, Daniel (DESY); ROLLES, Daniel (Kansas St. Univ.); ROMPOTIS, Dimitrios (DESY); SCHUBERT, Kaja (DESY); SCHWOB, Lucas (DESY); SENFFLEBEN, Björn (TU Berlin); TREUSCH, Rolf (DESY); ULMER, Anatoli (TU Berlin); ZIMBALSKI, Jannis (TU Berlin); ZIMMERMANN, Julian (TU Berlin); WEIGELT, Holger (DESY); MÖLLER, Thomas (TU Berlin); RUPP, Daniela (TU Berlin, MBI)*

Coherent diffractive imaging (CDI) at X-ray free-electron lasers allows for resolving the structure [1] and light-induced dynamics [2] in individual nanostructures. We developed an experimental setup saving two consecutive images on spatially separated detectors, allowing to follow the evolution of the same individual, non-reproducible particle after irradiation with an intense FEL pulse. In this context, we also developed and commissioned a multilayer-mirror-based split-and-delay unit (SDU) at FLASH delivering XUV-double pulses up to 650 ps delay [3]. The concepts of the imaging setup and the SDU will be introduced and proof-of-principle results will be presented.

[1] Rep. Prog. Phys, 80(11), 115901 (2017).

[2] New J. Phys., 18(4), 043017 (2016), Nat. Phot. 10, 93 (2016), Phys. Rev. Lett. 108, 093401 (2012).

[3] J. Synch. Rad., accepted (2018).



**The new URSA-PQ instrument for molecular photoenergy conversion at FLASH**

**Tuesday, 11:15  
MW1801  
ID 446**

***GUEHR, Markus (Institut für Physik und Astronomie, Universität Potsdam)***

*METJE, Jan (Institut für Physik und Astronomie, Universität Potsdam)*

We present the new URSA-PQ (Ultraschnelle Roentgenspektroskopie zur Abfrage der Photoenergiekonversion in Quantensystemen) instrument for the FLASH free electron laser in Hamburg. The instrument design is versatile and many different sources as well as detectors can be inserted, as the chamber adheres to CAMP instrument standard sizes. In its base version, a vacuum chamber with a magnetic bottle electron spectrometer, also capable of measuring ions, presents the main detector. A capillary oven will evaporate a solid molecular target creating a high local pressure at the interaction region of an optical pulse and the free electron laser x-ray pulse. The instrument is ready for user beamtime from second half of 2019 on and its progress can be followed on <http://exp-quantum.org/ursa-pq>.



**Atomic and Molecular Physics Experiments with the REMI Endstation at FLASH2**

**Tuesday, 11:30  
MW1801  
ID 443**

***MOSHAMMER, Robert (MPI für Kernphysik)***

A multi-particle coincidence spectrometer for electrons and ions (Reaction-Microscope, REMI) has been installed at the XUV free-electron laser FLASH2 in Hamburg as a permanent endstation for measurements with dilute atomic and molecular targets. The station is equipped with a highthroughput (> 70%) split-delay and focusing optics for the FEL beam in front of the spectrometer that allows XUV-pump – XUV-probe experiments with femtosecond resolution (defined by the FEL pulse duration) at high intensities (several  $10^{15}$  W/cm<sup>2</sup>). In addition, the newly installed shortpulse IR pump-probe laser at FLASH2 can be used for XUV-IR type excitation schemes. Moreover, in collaboration with the University Hannover and DESY a separate XUV source based on highharmonic generation is presently set up that will further extend the range of applications of the REMI station towards two-colour XUV pump-probe or transient absorption measurements. Besides these essentially technical aspects possible future applications as well as first results will be presented.



**Time-resolved studies of small molecules employing coincidence detection techniques**

**Tuesday, 11:45  
MW1801  
ID 441**

***JAHNKE, Till (IKF / Frankfurt University)***

With the implementation of the European x-ray free electron laser a unique tool for time-resolved studies of small molecules and clusters emerges. The targeted pulse durations and photon energies will enable investigations which, firstly, involve more tightly bound K-shell electrons, and, secondly, address typical timescales of molecular decay process in the low fs-regime. By means of coincidence detections techniques as, for example, Cold Target Recoil Ion Momentum Spectroscopy (COLTRIMS) it was demonstrated in the past, that in a few special cases, the molecular time domain is already accessible in measurements using synchrotron radiation. The talk will give a brief introduction to the topic and present two examples of such time-resolved coincidence measurements.



**New insights into ultrafast phenomena of solids by time-resolved spectroscopy with fs-XUV laboratory light sources**

**Tuesday, 12:00  
MW1801  
ID 435**

***STADTMÜLLER, Benjamin (Department of Physics and Research Center OPTIMAS, University of Kaiserslautern, Kaiserslautern, Germany)***

In condensed matter, the complex interactions between the subsystems spin, charge and lattice are responsible for (almost) all properties of solids. These interactions can lead to the formation of new phases of matter and determine the predominant energy and momentum relaxation mechanism of optically excited carriers in solids. In this regard, time-resolved (tr) pump-probe spectroscopy is an ideal tool to monitor the ultrafast dynamics of optically generated carriers and to correlate their relaxation pathway to the band structure of the solid. Recently, latest developments in fs-XUV light sources based on high-harmonic generation [1] have paved the way towards novel experimental capabilities. Here, I will discuss novel possibilities to study the ultrafast electronic and magnetic properties of solids by tr-absorption spectroscopy in reflection geometry and tr-and momentum resolved photoemission with fs-XUV radiation. These examples will show that these approaches can yield a novel comprehensive view onto ultrafast phenomena in solids.

[1] Popmintchev et al., Nat. Photon. 4, 822 (2010)



**Tuesday – September 18<sup>th</sup>, 2018**  
**11:00-12:30**

## ***Micro symposium 4***

### ***Industry and innovation***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>11:00-11:15</b> <i>Jan Vobecky</i> Innovation Concepts and Applications in Power-Electronics
<b>11:15-11:30</b> <i>Johannes von Borany</i> HZDR Ion Beam Center Activities for Industrial Collaboration and Innovation
<b>11:30-11:45</b> <i>Dmitry Doronkin</i> Operando XAS studies of direct synthesis of H <sub>2</sub> O <sub>2</sub> over Pd catalysts
<b>11:45-12:00</b> <i>Mariapaola Staropoli</i> Silica-filled SBR: filler structure evolution under quasi-static deformation
<b>12:00-12:15</b> <i>Ralph Gilles</i> Neutrons as a probe to study batteries in situ/operando
<b>12:15-12:30</b> <i>Marc Thiry</i> The SINE2020 Industry Consultancy and the role of neutron science in industrial innovation



**Innovation Concepts and Applications in Power-Electronics**

**Tuesday, 11:00  
MW0350  
ID 429**

***VOBECKY, Jan***

High energy ion beams are used in the power semiconductor industry since 1990s. They are used to prove the correctness of device design for sufficient cosmic ray withstanding capability under real device operation. Proton or helium beams are used to locally modify the excess carrier lifetime in order to widen the safe operation area of fast recovery diodes and switches in demanding application with fast high-power switches like IGBTs and IGCTs (Defect engineering). Proton beams have been also employed for modification of doping profiles by hydrogen donors at the end of wafer production, where high drive temperatures are not allowed anymore. All the cases above are illustrated by some design details of typical industrial products like diode, IGBT and thyristor. Companies without their own irradiation tools, like in the case of ABB Semiconductors, rely on the expertise and skills of engineers and researchers from large facilities. Some important aspects of this successful co-operation is also part of this contribution. A brief outlook into the future towards the wide bandgap semiconductors is given as well



## HZDR Ion Beam Center Activities for Industrial Collaboration and Innovation

Tuesday, 11:15  
MW0350  
ID 413

**VON BORANY, Johannes (Helmholtz-Zentrum Dresden-Rossendorf)**

*KLINGNER, Nico (Helmholtz-Zentrum Dresden-Rossendorf); AKHMADALIEV, Shavkat (Helmholtz-Zentrum Dresden-Rossendorf); KOLITSCH, Andreas (HZDR Innovation GmbH); BÖTTGER, Roman (HZDR Innovation GmbH)*

The HZDR Ion Beam Center (IBC) operates its facility as an international competence and user center for ion beam physics and applications providing all ions in an energy range between a few eV and almost 100 MeV. The available user beam time of about 14.400 h/a is shared between materials and interdisciplinary research work, but includes also a remarkable fraction of about 25% for collaborations and ion beam services to industrial partners. Collaborations with industry need specific conditions to foster bridges from research work to economic development and innovation and to enable sustainability. For that reason the ion beam services at IBC is delivered via the HZDR Innovation GmbH (HZDR spin-off from 2011) which cover all commercial, legal, quality management and liabilities issues. The presentation will illustrate some activities how industrial exploitation of ion beam technologies contributes to product innovations. As one example, in a close collaboration with the manufacturer an ion microscope has been qualified for elemental analysis of surfaces with a lateral resolution well below 100 nm using ion scattering or mass spectrometry techniques. Such a spatial analytical resolution marks a remarkable step for ion beam analysis imaging techniques. Another activity is the extensive application of high-energy implantation/irradiation techniques to realize novel devices for power- or opto-electronics which is of significant importance for requested energy saving electronics.



## Operando XAS studies of direct synthesis of H<sub>2</sub>O<sub>2</sub> over Pd catalysts

Tuesday, 11:30  
MW0350  
ID 424

**DORONKIN, Dmitry (KIT)**

*DESCHNER, Benedikt (Karlsruhe Institute of Technology); SHEPPARD, Thomas (Karlsruhe Institute of Technology (KIT)); SELINSEK, Manuel (Karlsruhe Institute of Technology); DITTMAYER, Roland (Karlsruhe Institute of Technology); GRUNWALDT, Jan-Dierk (KIT)*

Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is a widely used powerful, environmentally benign oxidizing agent. The liquid phase catalyzed direct synthesis of H<sub>2</sub>O<sub>2</sub> from molecular hydrogen and oxygen constitutes an attractive greener alternative to the industrial anthraquinone auto-oxidation process. However, insufficient mechanistic details hinder the development of enhanced catalysts and the choice of suitable reaction conditions for the direct synthesis of H<sub>2</sub>O<sub>2</sub>. Furthermore, safety concerns related to formation of explosive H<sub>2</sub>/O<sub>2</sub> mixtures severely restrict in situ / operando studies of the direct synthesis of H<sub>2</sub>O<sub>2</sub> as well as its commercial application. In order to gain insight in the catalytic processes occurring during the H<sub>2</sub>O<sub>2</sub> synthesis at the molecular level, we performed operando XAS studies of Pd catalysts in a continuous flow reactor at pressures of 10-40 bar. Water and ethanol were used as solvents. In both cases H<sub>2</sub>:O<sub>2</sub> ratio was varied while the complementary on-line product analysis allowed deriving structure-activity relationships and new insights into the mechanism of the direct H<sub>2</sub>O<sub>2</sub> synthesis [1].

[1] M. Selinsek, B.J. Deschner, D.E. Doronkin, T.L. Sheppard, J.-D. Grunwaldt, R. Dittmeyer, ACS Catal. 8 (2018) 2546.



**Silica-filled SBR: filler structure evolution under quasi-static deformation**

Tuesday, 11:45  
MW0350  
ID 270

**STAROPOLI, Mariapaola (Luxembourg Institute of Science and Technology)**

*PYCKHOUT-HINTZEN, Wim (Forschungszentrum Jülich); GERSTNER, Dominik (Goodyear S.A.); MEYER, Mathias (Goodyear S.A.); SCOLAN, Emmanuel (Luxembourg Institute of Science and Technology); WESTERMANN, Stephan (Goodyear S.A.)*

In this contribution, an investigation on the deformation mechanism of S-SBR/silica composites (0-90 phr of silica) by Small Angle Neutron Scattering (SANS) is presented. The study aims at a microscopic understanding of the filler aggregate structure and its evolution under uniaxial stress. A home-built stretching apparatus, employed at small-angle scattering instrument KWS-2 at MLZ in Garching, allowed in situ, time-resolved data acquisition during a programmed, quasi-static strain profile. 2D scattering patterns, for samples with filling degree above 60 phr, showed a butterfly-like, anisotropic pattern already in the undeformed state. The pattern became almost isotropic at intermediate strains ( $\epsilon \sim 20\%$ ). A change of the scattering pattern occurred at high strains ( $\epsilon \sim 60\%$ ), yielding a shape rotated by  $90^\circ$  compared to the pattern of the undeformed sample. The evolution of the anisometric scattering pattern with the applied deformation, could be explained by initial breaking-up of pre-oriented silica aggregates, promoted by occluded rubber inside. At higher strain, the compressing rubber matrix (perpendicular to strain) leads to additional alignment of latter smaller components. The alignment of fragmented clusters perpendicular to strain results in a rotation of the original pattern by  $90^\circ$ . An initial structural model to describe the scattering pattern evolution will be introduced in this contribution. Financial support by the FNR is acknowledged.



**Neutrons as a probe to study batteries in situ/operando**

**Tuesday, 12:00  
MW0350  
ID 338**

**GILLES, Ralph**

*SEIDLMAYER, Stefan; PAUL, Neelima; ZINTH, Veronika; VON LUEDERS, Christian (TU Muenchen ESS); BUCHBERGER, Irmgard (TU München TEC); HATTENDORFF, Johannes (TU Meunchen TEC); Dr WANDT, Johannes (TU München TEC); HOFMANN, Michael (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München); KUDEJOVA, Petra (TU München MLZ); SCHULZ, Michael; KNOCHE, Thomas (TU Muenchen iw); GASTEIGER, Hubert (TU Muenchen TEC); JOSSEN, Andreas (TU Meunechen ESS); Prof. REINHART, Gunther (TU Muenchen iw)*

In the last years neutron methods became very attractive for industry research on batteries. This contribution will give a few examples how neutrons support the industrial research on batteries using neutron diffraction, neutron imaging and prompt gamma activation analysis as methods of investigation.



**The SINE2020 Industry Consultancy and the role of neutron science in industrial innovation**

**Tuesday, 12:15  
MW0350  
ID 397**

***THIRY, Marc (Helmholtz-Zentrum Geesthacht )***

*BOUDOU, Caroline (Institut Laue-Langevin)*

How to develop an innovative process for efficient and high-quality development of advanced materials and components? Neutrons can see structures evolving in solid state materials, neutrons can see layers diffusing into each other, neutrons can assess the composition and stability of colloidal mixtures - with neutrons you can see materials from the inside, non-destructively, and while the step of interest in a manufacturing process is being taken. The EU-Horizon2020 project SINE2020 aims at strengthening the cooperation between industry and European neutron sources. Free of charge, the project offers training and education of industrial researchers and the possibility to perform test measurements at the participating neutron facilities. In this presentation we introduce the offer by the industry consultancy initiative in SINE2020 and show different neutron based analytical techniques we provided to industrial users on the basis of industry-relevant examples:

- Residual stress measurement with neutron diffraction
- Behaviour of a surfactant by small angle neutron scattering (SANS)
- Insights in fuel cells with neutron radiography
- Investigation of novel drug delivery systems by SANS
- and more

**Tuesday – September 18<sup>th</sup>, 2018**  
**14:00-15:30**

## ***Parallel session 1***

### ***Instrumentation and methods***

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

<p><b>14:00-14:15</b>  <i>Georg Rugel</i>            Running out of patience for radionuclide decay: Accelerator mass spectrometry (AMS) counts faster</p>
<p><b>14:15-14:30</b>  <i>Margarita Russina</i>            Upgrade Project of TOF spectrometer NEAT at Helmholtz Zentrum Berlin – new power for new science</p>
<p><b>14:30-14:45</b>  <i>Kristijan Kuhlmann</i>            Breakthrough in neutron backscattering spectroscopy: A tenfold enhanced energy resolution using GaAs</p>
<p><b>14:45-15:00</b>  <i>Mathias Richter</i>            Metrology with Synchrotron Radiation</p>
<p><b>15:00-15:15</b>  <i>Benjamin Bornmann</i>            In-situ and in-operando XAFS spectroscopy in the ms range at beamline P64 at PETRA III, DESY</p>
<p><b>15:15-15:30</b>  <i>Wolfgang Kreuzpaintner</i>            In situ Thin Film Growth for Polarized Neutron Reflectometry</p>



## Running out of patience for radionuclide decay: Accelerator mass spectrometry (AMS) counts faster

Tuesday, 14:00  
MW2001  
ID 325

**RUGEL, Georg (Helmholtz-Zentrum Dresden-Rossendorf)**

*MERCHEL, Silke (HZDR); LLOVERA, Roberto (Helmholtz-Zentrum Dresden- Rossendorf); SCHARF, Andreas (Helmholtz-Zentrum Dresden-Rossendorf); DREAMS-USERS*

Accelerator mass spectrometry (AMS) is the most sensitive analytical method to measure longlived radionuclides. Several AMS system has been recently established in Europe, however, most of them exclusively detecting carbon-14. At Dresden a 6 MV tandem accelerator is used for AMS since 2011: The DREAMS (DREsden AMS) facility [1-3] is part of the Ion Beam Center, a largescale user facility, where users apply for measurements via a proposal system. Radionuclides, which are routinely measured at DREAMS, are  $^{10}\text{Be}$ ,  $^{26}\text{Al}$ ,  $^{36}\text{Cl}$ ,  $^{41}\text{Ca}$ , and  $^{129}\text{I}$ . We recently also focussed on nuclides with shorter half-lives such as  $^7\text{Be}$  or  $^{44}\text{Ti}$ . The detection limits are generally several orders of magnitude better than any other mass spectrometry or decay counting method. AMS needs smaller sample sizes and measurements are finished within a few minutes to hours; though after performing chemical separation of the radionuclide from the sample matrix (ice, snow, rain, ground water, marine sediments, soil, meteorites, deep-sea nodules, lava, rocks). DREAMS users apply AMS to most diverse interdisciplinary projects. Prominent examples are the proof and dating of multiple supernovae during the last 10 Ma [4] and dating of a boulder from a rock fall triggered by a medieval Earthquake in the Nepal Himalaya [5].

[1] Akhmadaliev et al., NIMB 294 (2013) 5.

[2] Rugel et al., NIMB 370 (2016) 94.

[3] [www.dresden-ams.de](http://www.dresden-ams.de).

[4] Wallner et al., Nature 532 (2016) 69.

[5] Schwanghart et al., Science 351 (2016) 147.



## Upgrade Project of TOF spectrometer NEAT at Helmholtz Zentrum Berlin – new power for new science

Tuesday, 14:15  
MW2001  
ID 361

*RUSSINA, Margarita (Helmholtz Zentrum Berlin )*

*GÜNTHER, Gerrit (Helmholtz Zentrum Berlin ); GRZIMEK, Veronika (Helmholtz Zentrum Berlin ); DRESCHER, Lars (Helmholtz Zentrum Berlin ); KAULICH, Toralf (Helmholtz Zentrum Berlin )*

Neutron TOF spectrometer NEAT has a successful history of applications to the study of dynamics in the large time and length scale domains 0.01 – 100 ps and 0.05 - 5 nm, respectively, since the start of user operation in 1995. To address user community needs in more powerful instrumental capabilities, a concept of the full instrument upgrade has been developed and proposed. The upgrade started in 2010 as a HGF large scale project and the new instrument renew its user operation in January 2017. Thanks to the advanced design the upgrade resulted in 300 fold higher count rate compare to NEAT'1995 and in the instrument performance similar to the world leader IN5 at ILL, France despite an order of magnitude difference in the cold neutron flux. The features of the new instrument include novel integrated guide-chopper system, which 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 416 <sup>3</sup>He position sensitive detectors placed at 3 m from the sample and equipped by modern electronics using event recording data collection. The accessible wavelength range has been broadened into the thermal wavelength domain making NEAT a bispectral spectrometer and providing better conditions for low temperature studies. Non-magnetic instrument environment allows for studies with high magnetic fields up to 14 T and polarization applications. High count rate enables to reduce the mass of the sample from gm to mg. The upgrade makes NEAT'2016 one of the most advanced instrument worldwide and provides an outstanding experimental tool for broad range of research areas at BER II.



## Breakthrough in neutron backscattering spectroscopy: A tenfold enhanced energy resolution using GaAs

Tuesday, 14:30  
MW2001  
ID 241

**KUHLMANN, Kristijan (Friedrich-Alexander University Erlangen-Nürnberg / Institut Laue-Langevin)**

*APPEL, Markus (FAU Erlangen-Nürnberg / ILL); FRICK, Bernhard (Institut Laue-Langevin); MAGERL, Andreas (University Erlangen-Nürnberg)*

A prototype neutron backscattering spectrometer using the GaAs 200 Bragg reflection has been developed to bring about a substantial increase in energy resolution. Test measurements on the backscattering spectrometer IN16B at the ILL have yielded an unprecedented resolution of  $\delta E = 77$  neV FWHM [1], an order of magnitude smaller than the current standard of 750 neV FWHM as well as a factor of 4 smaller than the highest currently available resolution of 300 neV FWHM. In order to retain the high resolution offered by the narrow intrinsic line width of GaAs 200, several parameters need to be controlled to a high degree of precision, such as variations of the lattice parameter to  $(a/a) < 10^{-6}$ , temperature inhomogeneities to  $T < 0.3$  K over 10 m<sup>2</sup> of crystal surface, co-alignment of 4 × 4 mm<sup>2</sup> crystal facets to  $< 0.1^\circ$ , or mechanical strain to  $< 10^{-6}$ . Therefore, the prototype is modularised into small-scale components and equipped with a multichannel temperature control system. Further, we measured the hyperfine splitting in cobalt as a benchmark, providing proof of concept for the construction of a full-scale user instrument.

[1] K. Kuhlmann, M. Appel, B. Frick and A. Magerl, ILL Annual Report 2017, p. 82–83



## Application of multiple-orbit synchrotron radiation for electron time-of-flight spectroscopy

Tuesday, 14:45  
MW2001  
ID 331

**RICHTER, Mathias (Physikalisch-Technische Bundesanstalt)**

*ARION, Tiberiu; EBERHARDT, Wolfgang (Center for Free-Electron Laser Science at DESY); FEIKES, Jörg (Helmholtz-Zentrum Berlin); GOTTWALD, Alexander (Physikalisch-Technische Bundesanstalt); GOSLAWSKI, Paul (Helmholtz-Zentrum Berlin); KASER, Hendrik (Physikalisch-Technische Bundesanstalt); KOLBE, Michael (Physikalisch-Technische Bundesanstalt); LI, Ji (Helmholtz-Zentrum Berlin); LUPULESCU, Cosmin; RIES, Markus (Helmholtz-Zentrum Berlin); ROTH, Friedrich (TU Bergakademie Freiberg); RUPRECHT, Martin (Helmholtz-Zentrum Berlin); TYDECKS, Tobias; WÜSTEFELD, Godehard (Helmholtz-Zentrum Berlin)*

At the metrology light source (MLS), the compact electron storage ring of the Physikalisch-Technische Bundesanstalt (PTB) with a circumference of 48 m, a specific multiple orbit for stored electrons was realized closing after three turns. In combination with single electron bunch operation, the new mode was applied for electron time-of-flight spectroscopy with an interval of the synchrotron radiation pulses which is three times the single bunch period of revolution at MLS. The achievement is of significant importance for PTB's future programs of angular-resolved electron spectroscopy with synchrotron radiation and similar projects at other compact electron storage rings. Moreover, the applied scheme for orbit and source spot separation via optical imaging at the insertion device beamline of MLS can be considered partially as a proof of principle for BESSY VSR project of the Helmholtz-Zentrum Berlin.



**In-situ and in-operando XAFS spectroscopy in the ms range at beamline P64 at PETRA III, DESY**

**Tuesday, 15:00  
MW2001  
ID 347**

***BORNMANN, Benjamin (Bergische Universität Wuppertal)***

*KLÄS, Jonas (Bergische Universität Wuppertal); MÜLLER, Oliver (SSRL); WABNITZ, René (Uni Wuppertal); WAGNER, Ralph (Bergische Universität Wuppertal); LÜTZENKIRCHEN-HECHT, Dirk (Bergische Universität Wuppertal); FRAHM, Ronald (Bergische Universität Wuppertal)*

The beamline P64 at the high brilliance source PETRA III at DESY houses a dedicated quick-EXAFS monochromator for spectroscopy in the ms range and offers special equipment for in-situ and inoperando experiments. The monochromator consists of Si(111) and Si(311) crystals on an oscillating stage with frequencies of up to more than 50 Hz enabling spectra in the ms range. In the experimental hutch fast and custom-built gridded ionization chambers and amplifiers with precise ADCs are available to record the data with sufficient bandwidth continuously during experiments of up to a few hours. Lately, a 2-circle goniometer with linear table for reflectivity measurements was installed and commissioned. At incident angles between 0.1 and 0.8° the information depth can be varied between a few monolayers and some 10 nm. Additionally, a PIPS detector for fluorescence with a matched amplifier was tested at oscillation frequencies of up to 120 Hz. It can be used for surface sensitive experiments, for thicker samples where transmission is not possible, or for absorbing elements in concentrations down to 100 ppm or less. Several beamtimes with in-situ or in-operando catalysis experiments have been carried out successfully. The surface sensitive reduction of steel surfaces in different gas atmospheres at up to 1100°C was investigated in other experiments. All equipment is available for users, and the beamline is open for beamtime applications.



## In situ Thin Film Growth for Polarized Neutron Reflectometry

Tuesday, 15:15  
MW2001  
ID 2

**KREUZPAINTNER, Wolfgang** (*Technische Universität München, Physik-Dep. E21*)

*YE, Jingfan (Technische Universität München); BOOK, Alexander (Technische Universität München); INANLOO-MARANLOO, Zahra (Technische Universität München); HEIGL, Michael (Universität Augsburg); BÖNI, Peter (Technische Universität München); ALBRECHT, Manfred (Universität Augsburg)*

Magnetic films and heterostructures made of them or containing them are the basic building blocks of a large number of magneto-electronic devices whose fabrication is almost exclusively based on sophisticated thin film deposition techniques. The performance of the devices strongly relies on the magnetic properties of the layers they consist of. These are functions of the layers' morphology and microstructure and of the coupling between them. Since these parameters can change during the process of growth, it is highly desirable to analyse the development of the magnetic properties of heterostructures during the growth process and to correlate them with the structural parameters of the sample. While the in situ 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 and University Augsburg 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 current state in development will be shown, ranging from unpolarized and polarized proof of principle neutron reflectivity measurements on thin magnetic films carried out at the ToF reflectometer REFSANS at the FRM II neutron source to the latest fast in situ PNR measurements at the AMOR beamline at PSI. For the latter, the "Selene" neutron optical concept, based on elliptic neutron mirrors is essential. An overview over the latest developments and future modifications as well as the completion work, will also be given.



**Tuesday – September 18<sup>th</sup>, 2018**  
**14:00-15:30**

## ***Parallel session 8***

### ***Functional materials and materials science***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

**14:00-14:15**

*Stefan Seidlmayer*

Lithiation dynamics of graphite anodes investigated by in operando neutron diffraction

**14:15-14:30**

*Volodymyr Baran*

Neutron powder diffraction studies of prismatic Li-ion cell

**14:30-14:45**

*Neelima Paul*

Finding the lost lithium with neutron diffraction and physico-chemical modelling

**14:45-15:00**

*Susan Schorr*

Disorder in Zinc-Germanium Oxynitride: a neutron diffraction study

**15:00-15:15**

*Tobias Unruh*

Combined SAXS/SANS studies of functionalized interfaces

**15:15-15:30**

*Philipp Jordt*

Stress induced deformation in piezotronic micro structures studied by X-ray nano diffraction

## Lithiation dynamics of graphite anodes investigated by in operando neutron diffraction

Tuesday, 14:00  
MW0001  
ID 6

**SEIDLMEYER, Stefan**

ZINTH, Veronika; VON LÜDERS, Christian (Lehrstuhl für Elektrische Energiespeichertechnik (EES), TU München); WILHELM, Jörn (Lehrstuhl für Elektrische Energiespeichertechnik (EES), TU München); ERHARD, Simon (Lehrstuhl für Elektrische Energiespeichertechnik (EES), TU München); HATTENDORFF, Johannes (Lehrstuhl für Technische Elektrochemie (TEC), TU München); BUCHBERGER, Irmgard (Lehrstuhl für Technische Elektrochemie (TEC), TU München); GAN, Weimin; REBELO-KORNMEIER, Joana; HOFMANN, Michael; JOSSEN, Andreas (Lehrstuhl für Elektrische Energiespeichertechnik (EES), TU München); GILLES, Ralph

In lithium ion batteries the most commonly used anode material is graphite. The lithiation dynamics of graphite anodes is of crucial importance in respect to fast charging or low temperature charging of the cells, as well as aging processes can depend on the intrinsic lithiation properties. Commonly known issues have recently shown that even small effects can cause detrimental lithium plating and can impose a huge security issue. A fine understanding of the dynamic lithiation processes in commercial type cells is important to optimize the cell design and material properties in order to avoid security issues as well as unexpected performance deterioration due to fast aging. We have studied the lithiation dynamics at low and high temperature in commonly used 18650- type cells with in operando neutron diffraction and could show for the first time that intermittent Li plating can be observed even under not too harsh conditions. The Li plating has been elusive for a long time as our studies also reveal that the majority of the intermittently plated lithium is dissolved during very short relaxation times at room temperature and only remains for many hours at low temperature. The relaxation time is strongly dependent on temperature.

[1] Zinth, V., C. von Lüders, M. Hofmann, J. Hattendorff, I. Buchberger, S. Erhard, J. Rebelo- Kornmeier, A. Jossen and R. Gilles, 2014, Journal of Power Sources, 271, 152-159.

[2] Zinth, V., C. von Luders, J. Wilhelm, S. V. Erhard, M. Hofmann, S. Seidlmayer, J. Rebelo- Kornmeier, W. M. Gan, A. Jossen and R. Gilles, 2017, Journal of Power Sources, 361, 54-60.



## Neutron powder diffraction studies of prismatic Li-ion cell

Tuesday, 14:15  
MW0001  
ID 253

**BARAN, Volodymyr**

*MÜHLBAUER, Martin J. (Institute for Applied Materials (IAM), Karlsruhe Institute of Technology (KIT), Eggenstein-Leopoldshafen, Germany); SCHULZ, Michael; Mr PFANZELT, Joseph (Technical University of Munich, Research Neutron Source Heinz Maier-Leibnitz (FRM II), Garching, Germany); SENYSHYN, Anatoliy (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München)*

Li-ion batteries are playing an important role in powering portable devices, e-mobility applications and off-grid energy storage. Recently we showed that a neutron powder diffraction pattern taken on cylindrical cells like 18650-type can be successfully refined using a contribution from the positive and negative electrodes, current collectors and cell housing. Due to their rollover design the cylinder-type Li-ion cells are relatively difficult for lab manufacturing, i.e. contain numerous issues regarding reproducibility, current and electrolyte distribution etc. From the ergonomic point of view, the prismatic cells supply improved stacking/volumetric density, when compared to cylinder ones. This along with the increasing interest of cell manufacturers to the cells in prismatic shape in both low and high current applications inspired us to start the thorough characterization of the cells in this type using high-resolution neutron powder diffraction. In the current contribution three different approaches for the measurements of prismatic cells will be presented, i.e. fixed position, pseudo rotation and constant rotation. Besides this an application of rotation setup upon in situ/in operando conditions will be reported for the first time and compared to the behavior of 18650-type cell.

## Finding the lost lithium with neutron diffraction and physico-chemical modelling

Tuesday, 14:30  
MW0001  
ID 32

**PAUL, Neelima (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München (TUM),)**

*KEIL, Jonas; Mr KINDERMANN, Frank M.; Dr SCHEBESTA, Sebastian; DOLOTKO, Oleksandr (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München (TUM),); MUEHLBAUER, Martin; KRAFT, Ludwig; ERHARD, Simon V.; JOSSEN, Andreas; GILLES, Ralph*

Capacity fade in NMC/C 18650-type cells, cycled 1000 times at a 1C charge/discharge rate, has been characterized by in situ neutron diffraction and electrochemical analysis. Neutron diffraction of the cells show a cyclable lithium loss corresponding to a capacity fade of about 23% in both electrodes of the cycled cell, which is validated by electrochemistry. The cycled cell suffers an anode stoichiometry shift from  $x = 0.84$  to  $x = 0.65$  in  $\text{Li}_x\text{C}_6$  ( $0 \leq x \leq 1$ ) in its fully charged state and a cathode stoichiometry shift from  $y = 0.89$  to  $y = 0.81$  in  $\text{Li}_y(\text{Ni}_{0.33}\text{Mn}_{0.33}\text{Co}_{0.33})\text{O}_2$  ( $0 < y \leq 1.05$ ) in its fully discharged state. Anode ( $x = 0$ ) as well as cathode stoichiometries ( $y = 0.54$ ) remain practically unchanged in the cell's fully discharged and charged states, respectively. These experimental results have been validated by a physico-chemical aging model, which attributes capacity fade to loss of cyclable Li ions into the formation and growth of a continuous SEI film on the anode. The stoichiometry shifts extracted from neutron diffraction match well with those derived from the model, and both neutron diffraction and model are in good agreement to the electrically determined capacity fade of 21%. In fact, cyclable lithium losses slightly exceed this value. Thus, capacity fade in these cells is mainly due to loss of cyclable lithium into the continuous growth of a SEI film on the anode surface.

N. Paul, J. Keil, F. M. Kindermann, S. Schebesta, O. Dolotko, M. J. Mühlbauer, L. Kraft, S. V. Erhard, A. Jossen, R. Gilles, *Journal of Energy Storage* 17 (2018) 383-394.



## Disorder in Zinc-Germanium Oxynitride: a neutron diffraction study

Tuesday, 14:45  
MW0001  
ID 147

**SCHORR, Susan (Helmholtz-Zentrum Berlin für Materialien und Energie)**

*BRETERNITZ, Joachim (Helmholtz-Zentrum Berlin für Materialien und Energie); FRANZ, Alexandra (Helmholtz-Zentrum Berlin für Materialien und Energie)*

The ternary nitrides  $\text{ZnSnN}_2$  and  $\text{ZnGeN}_2$  have attracted attention as potential earth-abundant alternatives to III-V solar absorber materials [1]. In the aim to thoroughly understand the effect of oxygen inclusion on the structural features of those materials related to a number of synthesis conditions, we worked on the oxynitride system  $\text{Zn}_{1+x}\text{Ge}_{1-x}\text{O}_2\text{N}_{2-2x}$  ( $0 \leq x \leq 1$ ). The O<sup>2-</sup>/N<sup>3-</sup> charge difference is accounted for by an increase of the Zn<sup>2+</sup>/Ge<sup>4+</sup> ratio. In the ternary nitrides, cation ordering leads to a symmetry lowering from the wurtzite-type aristotype structure into a cation-ordered structure in Pna21. While the electronic similar elements Zn and Ge cannot be distinguished by conventional X-ray diffraction, neutron diffraction opens the possibility to clarify the question of cation order and, additionally, to differentiate between oxygen and nitrogen. Our neutron diffraction study, performed at the fine resolution neutron powder diffractometer E9 at BERII (HZB), showed that these oxynitrides adopt the wurtzite-type structure with disorder on both cation and anion sites. We will link our structural work with optical and chemical characterization in order to thoroughly understand this system that is, with a bandgap of  $E_g \approx 2.5$  eV, potentially suitable for wide-bandgap applications such as heterojunction solar cells.

[1] P. Narang et al., Adv. Mater., 2014, 26, 1235.

[2] Y. Lee et al., J. Phys. Chem. C, 2007, 111, 1042.



## Combined SAXS/SANS studies of functionalized interfaces

Tuesday, 15:00  
MW0001  
ID 151

*UNRUH, Tobias (FAU Erlangen-Nürnberg)*

*SCHMUTZLER, Tilo (FAU); LAGES, Sebastian (FAU); GÖTZ, Klaus (FAU); SCHINDLER, Torben (FAU)*

The combination of small angle X-ray and neutron scattering (SAXS, SANS) allows a detailed analysis of the mesoscopic structure of colloidal dispersions. It is, however, the atomic and molecular structure of the interface between the nanoparticles and the dispersion medium that governs in many cases the formation, stabilization, and function of the nanoparticles. In this contribution it will be demonstrated that it is possible to detect the details of stabilizing interface layers as e.g. the acetate layer of ZnO quantum dots in ethanolic solution, to discover and tailor the stabilizing mechanism of gold nanoparticles in aqueous solution crowded by CTAB micelles, and to observe the ligand (oleic acid – porphyrins) exchange and their molecular orientation at the interface of TiO<sub>2</sub> nanoparticles by simultaneous evaluation of SAXS and contrast variation SANS experiments. However, worldwide no instrument for simultaneous SAXS/SANS measurements is available so far. This limits the applicability of the combined analysis of SAXS and SANS data significantly especially for metastable systems. The first combined SAXS/SANS instrument which is currently under construction and will be installed in spring 2019 at the D22 instrument of the ILL (Grenoble, France) will be introduced briefly.



## Stress induced deformation in piezotronic micro structures studied by X-ray nano diffraction

Tuesday, 15:15  
MW0001  
ID 89

*JORDT, Philipp*

*MURPHY, Bridget; WARIAS, Jonas*

One promising approach to design bio magnetic field sensors is to combine piezoelectric and magnetostrictive materials in a magnetoelectric composite. Such sensors have the ability to generate an output in response to a magnetic signal from for example the human body. To achieve the extreme sensitivity of less than 100 pT required for medical applications, is a challenge. By using piezotronic readout from magnetoelectric sensors, it is possible to increase the limit of detection by three orders of magnitude. The piezotronic effect occurs in semiconductors with a non-centrosymmetric crystal structure. An induced piezoelectric potential causes additional piezoelectric charges at the metal-semiconductor interface resulting in a change of height and width of the Schottky barrier. The charge carrier transport across the metal-semiconductor contact is therefore dependent on the piezoelectric charges, which can be controlled by the magnitude and sign of the applied strain or vice versa. For this experiment we used ZnO micro wires with diameters between one and 100  $\mu\text{m}$ . We collected the electronic response of the sample and simultaneous applied a mechanical stress while observing key Bragg reflections. This nanofocus diffraction experiment provided a unique possibility to determine the spatially resolved lattice deformation in the device during piezotronic measurements.



**Tuesday – September 18<sup>th</sup>, 2018**  
**14:00-15:30**

## ***Micro symposium 3***

### ***Novel developments in time resolved techniques***

**MW1801**

*Each field is linked to the abstract's start page. Just click on the title!*

<b>14:00-14:15</b> <i>C. Sternemann</i> Combining x-ray emission and x-ray Raman spectroscopy at the scientific instrument FXE of the European XFEL
<b>14:15-14:30</b> <i>Peter Gaal</i> Ultrafast Bragg switch for hard X-rays
<b>14:30-14:45</b> <i>Ingo Uschmann</i> High Purity X-ray polarimetry @ PETRA III and EU X-FEL
<b>14:45-15:00</b> <i>Bridget Mary Murphy</i> Investigating structures and dynamics at liquid interfaces with ultra fast timescales
<b>15:00-15:15</b> <i>Benjamin Grimm-Lebsanft</i> Time-resolved X-ray absorption fine structure spectroscopy measurements at PETRA III beamlines P11 and P64
<b>15:15-15:30</b> Discussions



## Combining x-ray emission and x-ray Raman spectroscopy at the scientific instrument FXE of the European XFEL

Tuesday, 14:00  
MW1801  
ID 445

**STERNEMANN, C. (Fakultät Physik / DELTA, TU Dortmund, Dortmund, Germany)**

*BRESSLER, C. (European XFEL, Hamburg, Germany); TOLAN, M. (Fakultät Physik / DELTA, TU Dortmund, Dortmund, Germany); BIEDNOV, M. (European XFEL, Hamburg, Germany); ELBERS, M. (Fakultät Physik / DELTA, TU Dortmund, Dortmund, Germany); GALLER, A. (European XFEL, Hamburg, Germany); GAWELDA, W. (European XFEL, Hamburg, Germany); HARDER, M. (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany); KHAKHULIN, D. (European XFEL, Hamburg, Germany); KUBICEK, K. (European XFEL, Hamburg, Germany); LIMA, F.M. (European XFEL, Hamburg, Germany); OTTE, F. (European XFEL, Hamburg, Germany); SPIEKERMANN, G. (Institute of Earth and Environmental Science, Universität Potsdam, Potsdam, Germany); WEIS, C. (Fakultät Physik / DELTA, TU Dortmund, Dortmund, Germany); WILKE, M. (Institute of Earth and Environmental Science, Universität Potsdam, Potsdam, Germany); ZALDEN, P. (European XFEL, Hamburg, Germany)*

X-ray emission and x-ray Raman scattering spectroscopy are powerful methods to study in situ the local electronic and atomic structure of high and low Z elements, respectively, in bulk matter. A combination of these techniques for time-resolved experiments should deliver a deeper understanding of phenomena such as e.g. hydration shell dynamics or the correlation between spin and structural dynamics. The scientific instrument FXE at the European XFEL provides unique experimental capabilities for simultaneous K $\beta$  and valence to core emission, as well as x-ray Raman scattering spectroscopy, on picosecond to femtosecond timescales using a wavelength dispersive von Hamos spectrometer together with a 6-element Johann-type spectrometer. We discuss the current status of the setup and present a short outlook on future experiments.



## **Ultrafast Bragg switch for hard X-rays**

**Tuesday, 14:15**  
**MW1801**  
**ID 156**

***GAAL, Peter (Universität Hamburg)***

*SANDER, Mathias (European Synchrotron Radiation Facility); BAUER, Roman (Universität Hamburg)*

We present a time-resolved ultrafast x-ray diffraction experiment with 5-10 ps temporal resolution performed at ID09B beamline at ESRF. The synchrotron hard x-ray pulse with an original duration of 120 ps is shortened by an ultrafast photoacoustic Bragg switch. The short x-ray pulse is used to probe lattice dynamics in a double-layered thin film sample. We discuss in detail the quality and design parameters of the device, such as switching contrast, switching efficiency and structural parameters and analyze changes in Brilliance due to introduction of the Bragg switch into the beamline.



### High Purity X-ray polarimetry @ PETRA III and EU X-FEL

Tuesday, 14:30  
MW1801  
ID 320

*USCHMANN, Ingo (Friedrich-Schiller-Universität Jena)*

*ROEHLBERGER, Ralf (Deutsches Elektronen-Synchrotron DESY); WILLE, Hans-Christian (DESY); LOETZSCH, Robert (Helmholtz-Institut Jena; Institute of Optics and Quantum Electronics)*

High purity X-ray polarimetry has become a powerful method at x-ray sources of third and fourth generation as well as innovative laboratory sources. Their application has been extended to nuclear resonant scattering, quantum optics in the x-ray range, spectro-polarimetry at selected absorption edges as well as vacuum quantum electrodynamic. The installation of high purity polarimeter allows users at PETRA III beam line P01 and at EU X-FEL beam line MID to use polarimeters which consist of polarizer and analyzer based on multiple reflection channel cut crystals providing a polarization selection up to 10 orders of magnitude in intensity. The physical limits of polarization purity will be given in the contribution. By selecting perfect or nearly perfect crystals of different atomic numbers one can favor the purity or transmitted intensity. Different crystals such as silicon, germanium, or diamond could yield purities in the range between  $10^{-10}$  ...  $10^{-7}$ . The instruments located at the synchrotron and FEL will be described. Application experiments in the field of quantum optics such as vacuum-assisted generation and control of atomic coherences at x-ray energies, tunable subluminal propagation of narrow-band X-ray pulses, and collective strong coupling of X-rays and nuclei in a nuclear optical lattice will be presented by using nuclear resonant scattering of the  $^{57}\text{Fe}$  isotope.



## Investigating structures and dynamics at liquid interfaces with ultra fast timescales

Tuesday, 14:45  
MW1801  
ID 437

*MURPHY, Bridget Mary (Kiel University)*

Liquid interfaces witness renewed interest from physics, chemistry, and biology to gain fundamental insight and to develop applications, including nanomaterial synthesis and food science. To extend investigations of structure and growth at liquid – vapour[1] and liquid – liquid interfaces[2] to ultrafast time scales, we recently installed an optical pump – X-ray probe facility at the Liquid Interfaces Scattering Apparatus (LISA) [3]. The LISA diffractometer, at the P08 beamline at PETRA III, is specialized for X-ray scattering studies of liquid interfaces without moving the sample. In the new development, a synchronized femtosecond-laser system and optics direct a laser pulse onto the liquid sample surface delivering a time resolution better than 100ps. These new capabilities provide access to structural changes on nanometer length scales induced via optical excitation, allowing us to understand the non-equilibrium processes on liquid interfaces. In the case of liquid mercury, we focus on non-equilibrium dynamics of capillary waves after ultrafast thermal excitation. We have also investigated pure water and water-based solutions to investigate the influence of excited solvated electrons and their relaxation on the structure of the free liquid surface. We thank the BMBF (05K13FK2) for funding.

1. J. Haddad, et al. PNAS 201716418 (2018).
2. B. M. Murphy, et al., Nanoscale 8, 13859 (2016).
2. B. M. Murphy et al., J.Synchrotron Rad. 21, 45 (2014).



## Time-resolved X-ray absorption fine structure spectroscopy measurements at PETRA III beamlines P11 and P64

Tuesday, 15:00  
MW1801  
ID 434

**GRIMM-LEBSANFT, Benjamin (Institut für Nanostruktur- und Festkörperphysik, Universität Hamburg)**

*DICKE, Benjamin (Universität Hamburg, Institut für Nanostruktur- und Festkörperphysik); HOFFMANN, Alexander (Institut für Anorganische Chemie, RWTH Aachen); STANEK, Julia (Institut für Anorganische Chemie, RWTH Aachen); NAUMOVA, Maria (DESY); BIEBL, Florian (Institut für Nanostruktur- und Festkörperphysik, Universität Hamburg); TEUBNER, Melissa (Institut für Nanostruktur- und Festkörperphysik, Universität Hamburg); BIEDNOV, Mykola (Institut für Nanostruktur- und Festkörperphysik, Universität Hamburg); CALIEBE, Wolfgang (DESY); HERRES-PAWLIS, Sonja (Institut für Anorganische Chemie, RWTH Aachen); RÜBHAUSEN, Michael (Institut für Nanostruktur- und Festkörperphysik, Universität Hamburg)*

We present the results of our time-resolved X-ray absorption fine structure spectroscopy (TR-XAS) measurements at PETRA III beamline P11 and our future plans for TR-XAS measurements at beamline P64. With time-resolved pump-probe measurements using a high intensity micrometer-sized X-ray beam on copper complexes with a specifically designed constraining or predistorted ligand geometry, also called entatic state, we found metal-to-ligand charge-transfer state lifetimes that are very short. The entatic state denotes a distorted coordination geometry of a complex from its typical arrangement that generates an improvement to its function. The entatic-state principle has been observed to apply to copper electron-transfer proteins and it results in a lowering of the reorganization energy of the electron-transfer process. Here it is transferred to photoactive complexes. Additional Raman, TR-Fluorescence and TR-UV/Vis measurements also show the same time scales making them ideal complementary tools. Future plans are to implement a time-resolved XAS setup at beamline P64 to investigate Tyrosinase model complexes. Static measurements have already been performed and show a very promising perspective.



**Tuesday – September 18<sup>th</sup>, 2018**  
**14:00-15:30**

## ***Micro symposium 5***

### ***Digital agenda***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

**14:00-14:30**

*Hans Fangohr*

Digital Agenda – challenges and opportunities for data analysis

**14:30-15:00**

*Jonathan Taylor*

Scientific software development and data management at the European Spallation Source

**(no abstract available)**

**15:00-15:30**

*Martin Boehm*

Neutron experiments with virtual access



**Digital Agenda – challenges and opportunities for data analysis**

**Tuesday, 14:00-14:30**  
**MW0350**  
**ID 452**

***FANGOHR, Hans (European XFEL, University of Southampton)***

The discussion of the Digital Agenda attracts lots of attention to potential challenges and opportunities for science. In this presentation I want to focus on these aspects for computational science and data science. I will address the question of reproducibility of scientific results, and the related question of re-usability of published results and re-use of data sets. With some focus on data analysis of large data sets from research facilities, I will also discuss data policy, scientific, technical and social challenges that we need to address.



### Neutron experiments with virtual access

Tuesday, 15:00-15:30  
MW0350  
ID 454

**BOEHM, Martin (Insitut Laue-Langevin)**

*MUTTI, Paolo (Institut Laue-Langevin); WEBER, Tobias (Institut Laue-Langevin); LE GOC, Yannick (Institut Laue-Langevin)*

Today's user community appreciates the active hand-on participation in neutron experiments and the scientific exchange at the facilities, which act as international crossings for experts of very different scientific fields. This user program, on the other hand, imposes relatively long missions and puts time constraints on participants and instrument schedules, which hinders flexible handling of experiments or rapid integration of hot scientific topics. In future, a major severe constraint to the present user access arises from changing legislation, which has to assure increasing security standards for protecting nuclear installations and/or for protecting against misappropriation of materials on-site. With NEVA we explore an alternative, virtual, access mode for neutron experiments. We merge existing instrument control software, computing and scientific software tools, state-of-the-art communication technology and modern 3D animation into a virtual interactive platform, accessible on-site or remotely. NEVA will be open source and platform independent and usable with different instrumental techniques at the different facilities. The software will be a modern web application written in fast, just-in-time compiled Javascript.



**Tuesday – September 18<sup>th</sup>, 2018**  
**16:00-17:30**

## **Poster session 2**

<b>ID</b>	<b>Title</b>	<b>Presenter</b>
1	The Engineering Diffractometer BEER at ESS: A Status Update	<i>Jochen Fenske</i>
5	Molecular properties and growth conditions associated with PSS diffusion during annealing in polyelectrolyte multilayers	<i>Annekatriin Sill</i>
8	Modern diffraction methods for the investigation of thermo-mechanical processes	<i>Klaus-Dieter Liss</i>
9	Novel Reflection High-Energy Positron Diffractometer at NEPOMUC	<i>Matthias Dodenhoeft</i>
10	Nanoscale cavitation study in a laval nozzle by saxs	<i>Guenter Rinke</i>
11	Uncover processes at the interface with neutrons	<i>Henrich Frielinghaus</i>
12	Ab-initio methods for membrane protein structure recovery from small-angle neutron scattering data	<i>Alexandros Koutsioumpas</i>
13	Two for the price of one (proposal): MBE sample growth and polarised neutron reflectometry at MLZ	<i>Sabine Pütter</i>
14	The high intensity reflectometer of the JCNS: MARIA	<i>Alexandros Koutsioumpas</i>
16	Interface effects in superconductor-ferromagnet heterostructures	<i>Annika Stellhorn</i>
18	Self-organization of shape anisotropic nanoparticles at the liquid-air interface	<i>Flore Mees</i>
19	Interlayer correlations in 1:1 ferecrystals	<i>Maria Hentschel</i>
20	Single and double layered square arrays of magnetic nanoparticles	<i>Dominique Dresen</i>
21	Topologically stable helices in exchange-coupled rare-earth/rare-earth multi-layer with superspin-glass-like ordering	<i>Jingfan Ye</i>
24	Proximity effects across oxide interfaces of superconductor-insulator-ferromagnet hybrid heterostructure	<i>Amitesh Paul</i>
27	Structural and magnetic properties of cobalt iron disulfide nanocrystals	<i>Neelima Paul</i>
31	Aging studies and influence of anode in LiFePO <sub>4</sub> -based cells with neutron diffraction	<i>Neelima Paul</i>
38	JCNS @ ILL: News and progress on IN12	<i>Wolfgang Schmidt</i>
40	Time-resolved X-ray diagnostics of pulsed laser ablation in liquids (PLAL)	<i>Anton Plech</i>
42	Grating-based phase-contrast microtomography at PETRA III	<i>Felix Beckmann</i>
43	Voltage control of magnetism in oxide heterostructures: Neutron & X-ray and electron microscopy investigation	<i>Tanvi Bhatnagar</i>
44	The Structural and magnetic properties of ordered arrangements of magnetic nanoparticles	<i>Asmaa Qdemat</i>
46	Morphology and crystallinity of Sr <sub>x</sub> Co <sub>y</sub> O <sub>z</sub> films at different growth conditions and stoichiometry	<i>Patrick Schöffmann</i>



47	High-pressure crystallographic studies in diamond anvil cells using neutrons at HEIDI	<i>Andrzej Grzechnik</i>
63	Frustration in Sm-based pyrochlores	<i>Viviane Peçanha Antonio</i>
64	Combination of kinetic and structural studies of catalysts at the CAT-ACT X-ray spectroscopy beamline at KIT	<i>Anna Zimina</i>
66	Magnetic properties and lattice dynamics of $Mn_3Fe_2Si_3$ single crystal	<i>Mohammed Haddouch</i>
67	KWS-1 SANS instrument with polarization analysis	<i>Artem Feoktystov</i>
69	The New Small-Angle Neutron Scattering Instrument SANS-1 at MLZ – Features and First Results	<i>Sebastian Muehlbauer</i>
72	Pseudo-Goldstone Magnons in the Frustrated S=3/2 Heisenberg Helimagnet $ZnCr_2Se_4$ with a Pyrochlore Magnetic Sublattice	<i>Yevhen Onykiienko</i>
74	Morphologies and Solvent Distribution During Solvent Vapor Annealing of Block Copolymer Thin Films: In situ, Real-time GISAXS Investigations	<i>Christine Papadakis</i>
77	In situ GISAXS Investigations of pH and Temperature Responsive Block Copolymer Thin Films during Swelling in Water Vapor	<i>Florian Jung</i>
78	New developments on the SANS instrument D11 at the ILL	<i>Ralf Schweins</i>
79	Impact of interfacial solvent restructuring onto catalytic behavior by PDF	<i>Mirco Eckardt</i>
81	Hard X-ray spectroscopy of magnetic thin films for spintronic devices	<i>Andrei Gloskovskii</i>
82	Directional, hierarchical films via spray coating	<i>Julian Heger</i>
83	The anomalous breakdown of the Stokes-Einstein relation in Ge-Sb-Te and Ag-In-Sb-Te alloys and its connection to fast crystallization in the super-cooled liquid	<i>Shuai Wei</i>
84	Spin Structure in Magnetic Nanospheres	<i>Dominika Zákutná</i>
86	A compact and calibratable von Hamos X-Ray Spectrometer based on two full-cylinder HAPG mosaic crystals for high-resolution XES and RIXS	<i>Ina Holfelder</i>
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## The Engineering Diffractometer BEER at ESS: A Status Update

ID 1

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The time-of-flight engineering diffractometer BEER currently under construction at the European Spallation Source (ESS) is dedicated to the support of the latest progress in development, fabrication and optimization of modern engineering materials by neutron scattering analysis. The main tasks of BEER are to enable fast in situ and in operando characterization of materials and their microstructure during processing conditions close to real ones and to provide state-of-the-art and fast analysis of residual stresses, microstructure/crystallographic texture characterisation and phase analysis. These tasks are supported by a newly developed chopper technique called pulse modulation. It extracts several short pulses out of the long ESS pulse. Thus leading to a multiplexing of Bragg reflections and to substantial intensity gain for high symmetric materials while preserving the resolution. By the combination of the new modulation technique with a standard pulse shaping technique, BEER is a versatile engineering diffractometer providing easy tuneable resolution/flux ratios across wide wavelength and resolution ranges. Together with a large detector coverage, BEER enables sub-second in situ measurements for fast residual strain scans; texture analysis as well as phase analysis of complex composite systems where high resolution is needed. Here, we present an update of the BEER instrument design, its features and the current progress of the construction.

## Molecular properties and growth conditions associated with PSS diffusion during annealing in polyelectrolyte multilayers

ID 5

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During annealing of polyelectrolyte multilayers in concentrated solutions (1 M NaCl) interdiffusion of polyelectrolytes occurs. We investigate the interdiffusion perpendicular to the substrate using neutron reflectivity and selectively deuterated polyanions (PSS). Multilayers formed at 10 mM NaCl consist of flatly adsorbed chains. The diffusion constant of PSS can be tuned by four orders of magnitude; it decreases exponentially with the degree of polymerization  $N$  of PDADMA. Multilayers formed at 100 mM NaCl consist of interdigitated chains. When the degree of polymerization of PDADMA exceeds the one of PSS, the diffusion constant drops suddenly by three orders of magnitude and remains low. Such sudden transitions are better known from network than from polymer theory.



## Modern diffraction methods for the investigation of thermo-mechanical processes

ID 8

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The development, processing and functionality of metals and alloys is a fine-tune between atomic structure, micro-structure and mechanical properties of a final product. In-situ neutron and synchrotron X-ray diffraction deliver unique and complementary insight into the microstructural evolution of metals under various conditions, such as at high or low temperature, pressure and plastic deformation. Neutrons illuminate a larger bulk volume and reveal quantitative phase abundance, bulk texture, lattice parameter changes and other ensemble averaged quantities. Scattering contrasts complementary to X-rays can reveal atomic disorder in ordered intermetallics, particularly titanium alloys, while the dynamical theory of diffraction has been employed to study their defect kinetics at very high temperature. In contrast, fine-bundled high-energy X-rays deliver reflections from a number of individual grains. For each constituting phase, their statistics and behavior in time reveal information about grain growth or refinement, subgrain formation, static and dynamic recovery and recrystallization, slip systems, twinning, etc. A review on example systems is given with an outlook to the Materials Oscilloscope.

## Novel Reflection High-Energy Positron Diffractometer at NEPOMUC

ID 9

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The precise knowledge of the surface structure is essential to understand e.g. chemical reactions, optimize catalytic techniques or develop nanoelectronic devices. It has been shown that Total Reflection High-Energy Positron Diffraction (TRHEPD) is an ideal technique to determine the structure of the topmost and the immediate subsurface atomic layer of crystals [1]. In contrast to electrons, which are used in Reflection High-Energy Electron Diffraction (RHEED), positrons are repelled by the crystal potential and exhibit the phenomenon of total reflection for small angles of incidence. For this reason, TRHEPD features outstanding surface sensitivity and thus provides information, which cannot be obtained with conventional techniques such as RHEED or SXRD. Currently, we set up a new  $e^+$  diffractometer that will be coupled to the high-intensity positron source NEPOMUC [2]. After commissioning in 2018, first  $e^+$  beam experiments will be performed on reference samples such as Si(111) and Ge(110) and with various  $e^+$  energies (10-30keV) to demonstrate the superior features of the new diffractometer. This project is financially supported by the BMBF (funding number 05K16WO7).

[1] I. Mochizuki et al., Phys. Chem. Chem. Phys. 18, 7085 (2016)

[2] C. Hugenschmidt et al., J. Phys. Conf. Ser. 505 012029 (2014)

## Nanoscale cavitation study in a laval nozzle by SAXS

ID 10

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Fluid transport can suffer from phenomena that are known in cavitation around propeller blades. The underlying nucleation phenomena, like heterogeneous nucleation due to surface roughness or impurities in the bulk phase, are known. Up to now only results of measurements on a later stage of bubble formation using fast video photography and synchrotron imaging have been published. We used SAXS at the mySpot beamline (BESSY II) to investigate the onset of nanoscale fluctuations, developing sub-micron sized bubbles in a Laval nozzle, in which water is flowing at controlled pressure (up to 1 bar) and throughput. The Laval nozzle consists of a constriction of 5 mm in a slotted 3 mm thick metallic plate enclosed between two silicon plates, which transmit the 19 keV x-ray beam. The maximum velocity of water at the narrowest position amounts to 20 m/s. We used a closed-loop arrangement with a water reservoir, a pump and the Laval nozzle. The scattering pattern is mapped in a spatial cross section through the nozzle. Different suspensions of SiO<sub>2</sub> nanoparticles were used with particle sizes of 20 – 30 nm as possible seeds for cavitation. The concentration of nanoparticles had a range between 10<sup>7</sup> and 10<sup>9</sup> per cm<sup>3</sup>. Scattering distribution across the nozzle cross section is mapped and correlated to mechanisms of heterogeneous nucleation (on nanoparticles and on walls) as well as on homogeneous nucleation, which is difficult to discern.



## Uncover processes at the interface with neutrons

ID 11

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The method grazing incidence small angle neutron scattering (GISANS) is about having a breakthrough, but one essential step needs to be taken: We need a dedicated GISANS instrument at the MLZ that allows to monitor processes. Neutrons have utmost advantages over x-rays: We can monitor processes at the solid-liquid interface that is nearly impossible for x-rays. In many applications we need to follow structural changes during the operation of the solid-liquid interfaces such as in batteries, in fuel cells, and in catalytic reactions. Therefore, we need a dedicated instrument that focuses on the Q-range of interest, and monitors the process in shortest time-slices. This needs a new dedicated GISANS instrument that is not available anywhere in the world. From existing instruments (REFSANS, MARIA, and KWS1), which are less optimized for GISANS measurements, we can guess that shortest time-slices of 10 to 30 minutes are possible, if all parameters of the new instrument are optimized. This would bring new quality to surface science, which so far was dominated by the x-ray community. Other hot topics where neutrons introduce a new or varying contrast are (a) organic and hybrid solar cells, (b) the deposition process of films and coatings, and (c) magnetic and non-magnetic particles. In combinations with x-rays, new multicomponent structures can be resolved in more detail, and specific roles of particular components discovered. It is a strategy of the new GISANS instrument at the MLZ to make use of synergies with other instruments at the ESS, at DESY and the ALS to proceed even faster with software development, data analysis, and scientific output. The experts of the instruments and the exchanging user community will maximize the output of the linked instrument suite.



## Ab-initio methods for membrane protein structure recovery from small-angle neutron scattering data

ID 12

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The study of membrane protein structure by high resolution techniques has proven to be quite challenging and only recently with the development of modern cryo-electron microscopy methods, associated difficulties seem to become partially addressed. Different approaches have been developed for solubilizing membrane proteins while at the same time keeping them folded and active involving the use of detergents that shield the hydrophobic transmembrane surface of the protein which otherwise leads to the aggregation of the protein in solution. Success in keeping membrane proteins stable in solution permits the use of small-angle scattering (SAS) for the investigation of the protein/detergent complexes. However till today only a limited number of published works have attempted the use of SAS for related studies mainly due to the lack of theoretical tools for the treatment of scattering data for these particular systems. Recently we have presented an ab-initio method [Biophys. J. 113, 2373] based on multi-contrast SANS for the determination of the low-resolution structure of detergent-solubilized membrane proteins. It has been shown that two contrast SANS measurements appear to provide enough information for obtaining reliable shape reconstructions. In this contribution we aim to review the methodological aspects of this approach, showcase aspects of its application in a range of different protein / detergent systems and also discuss its complementarity to other methods.

## Two for the price of one (proposal): MBE sample growth and polarised neutron reflectometry at MLZ

ID 13

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Imagine you have an idea for a thin film system which you want to measure with polarised neutron reflectometry. But you do not have the expertise and/or the equipment for thin film fabrication. We can help you! The JCNS thin film laboratory runs an oxide MBE system for the growth of various types of samples, i.e. “classical” magnetic thin films, transition metal oxide heterostructures or thin metal films for soft matter studies, acting as defined surfaces. Access to the thin film laboratory is provided through application for beam time at a neutron instrument, e.g. the polarised neutron reflectometer MARIA. Simply, the MBE system access application has to be added to the proposal. This form asks for the specifications of sample preparation ([www.mlz-garching.de/mbe](http://www.mlz-garching.de/mbe)). In the presentation, we will give an overview for high quality metal and complex oxide thin film systems all fabricated in the JCNS thin film laboratory, like SrCoO<sub>x</sub>, La<sub>1-x</sub>Sr<sub>x</sub>MnO<sub>3</sub>, Fe<sub>4</sub>N or Co/Pt multilayers. The focus lies on stoichiometry, morphology and thickness precision. Detailed information about the possibilities of sample fabrication for users will be given. For quasi in-situ neutron reflectometry of thin films, which are sensitive to ambient air, a small versatile transfer chamber is offered for sample transfer from the MBE laboratory to the neutron instrument MARIA and measurement under UHV conditions.

## The high intensity reflectometer of the JCNS: MARIA

ID 14

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The MAGnetism Reflectometer with high Incoming Angle (MARIA) is a world class vertical sample reflectometer dedicated to the investigation of thin films and interfaces in the field of magnetism, material science, soft matter and biology. The elliptic vertically focusing guide allows the investigation of small samples with a typical size of  $1 \times 1 \text{ cm}^2$  quite efficiently. The double bounce polarizer and the in-situ pumped  $^3\text{He}$  SEOP neutron spin filter cell for analyzing the polarization of the reflected neutron beam can be moved into the beam in seconds. The polarized flux of MARIA amounts to  $5 \cdot 10^7 \text{ n}/(\text{s} \cdot \text{cm}^2)$  at the sample position with a horizontally collimated beam of  $3 \text{ mrad}$  and a wavelength of  $\lambda = 0.45 \text{ nm}$  with a wavelength resolution of  $\Delta\lambda/\lambda = 10\%$ . In the nonpolarized mode a flux of  $1.2 \cdot 10^8 \text{ n}/(\text{s} \cdot \text{cm}^2)$  is reached in this configuration. MARIA can be also operated in Grazing Incidence Small Angle Neutron Scattering (GISANS) mode by forming a pinhole collimation with the two 4 segment slits and an absorber preventing the focusing of the elliptic guide in the vertical direction. In the present contribution we review the full range of available sample environments for measurements in high magnetic fields/cryogenic temperatures (hard matter) and the automatic solid/liquid cell sample changer for soft matter applications. Finally we focus on some scientific highlights from our user program.

## Interface effects in superconductor-ferromagnet heterostructures

ID 16

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At interfaces of ferromagnetic (F) and superconducting (S) layers proximity effects can lead to significant changes in their order parameters. When the magnetic state of the F-layer is inhomogeneous, magnetic domains can spatially confine the superconductivity in an adjacent S-layer [1]. Our goal is to obtain an understanding of such proximity effects between the two layers. Furthermore, the lateral magnetic depth profile near the S/F-interface and the dependence of the superconductivity on the magnetic configuration still have to be scrutinized. As a prototype system we use thin film heterostructures of ferromagnetic FePd with a superconducting Nb toplayer. The heterostructures are grown using molecular beam epitaxy. To obtain FePd in the L10-ordered phase with a magnetic anisotropy perpendicular to the surface plane, growth temperatures between 230°C-350°C are used [2]. Resistivity measurements as a function of an external magnetic field  $H$  reveal the effect of the magnetic stray fields on the superconducting state. When the superposition of the stray fields and  $H$  reaches its minimum, superconductivity nucleates over the domain with magnetization direction opposite to  $H$ . To investigate the depth profile of the lateral magnetization fluctuation Grazing-Incidence Small-Angle Neutron Scattering (GISANS) is performed near the superconducting critical temperature.

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## Self-organization of shape anisotropic nanoparticles at the liquid-air interface

ID 18

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Self-organization of inorganic nanoparticles at liquid/air interfaces is a promising approach towards fabrication of novel functional 2D materials [1]. For spherical nanoparticles, the formation of two-dimensional superlattices was reported [2,3], whereas for shape anisotropic nanoparticles the detailed mechanism of nanoparticle organization at liquid/air interfaces requires further insight. In-situ monitoring of the particle arrangement at the liquid/air interface during compression is a promising approach towards the lateral packing as well as the vertical orientation and contact angle. For our study of the shape-dependent nanoparticle self-organization at the liquid/air interface, we have synthesized monodisperse spherical and cubic maghemite nanoparticles and determined their particle size, shape and chemical composition. The samples were spread onto a H<sub>2</sub>O/D<sub>2</sub>O subphase using a Langmuir set-up, which enables tuning the mean interparticle distance by variable surface compression. Structural information on the self-organization process was obtained from neutron reflectometry (NR) measurements, carried out at REFSANS (MLZ), leading to enhanced understanding of the nanoparticle self-organization at different compression points.

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## Interlayer correlations in 1:1 ferecrystals

ID 19

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Understanding and controlling nucleation and growth is an important step in improving the crystallinity of thin films and thus crucial for the materials performance.<sup>1</sup> Nanolaminates represent ideal model systems to study interfacial nucleation, because they offer precise control on a sub-Å length scale. Ferecrystals are nanolaminate compounds with the general formula  $[(MX)_m]_m[TX_2]_n$  (M = Pb, Sn, Bi, or rare earth metal; X = S, Se, Te; T = transition metal,  $\delta$  the misfit parameter).<sup>2</sup> The individual ferecrystal components are precisely oriented in the stacking direction but generally rotationally disordered in plane<sup>2</sup> and can thus be regarded as individual nanostructures.<sup>3</sup> In contrast, recently studied  $m = n = 1$  ferecrystals indicate directionally dependent interlayer registration.<sup>4</sup> Here we present a systematic study of such interlayer structural correlations. Unexpected reflections are identified by reciprocal space mapping that indicate interlayer correlations between the binary compounds, appearing stronger with improved lateral lattice match. Our results provide important insight into the formation mechanism of ferecrystals, suggesting preferential nucleation and layer alignment during self-organization.

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3 M. Beekman, S. Disch, D. C. Johnson, et al. Angew. Chem. 52, 13211, (2013).

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## Single and double layered square arrays of magnetic nanoparticles

ID 20

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Ordered nanostructures of magnetic nanoparticles are interesting both due to potential applications, e.g. in information technology and spintronics [1, 2], and for the fundamental investigation of dipolar coupling [3, 4]. In particular stacked structures of ordered magnetic nanoparticle monolayers are interesting due to their potential for highly structured 3D devices and as model systems for the study of nanoscale magnetism. Here, we present our recent studies on the preparation and characterization of single domain magnetic nanoparticles arranged in a dense square lattice as monolayer and as double layer with tunable interlayer distance. The work presents an easy way to prepare high quality model systems of layered nanoparticle arrays. The nanostructures are characterized structurally by electron microscopy, grazing-incidence small-angle scattering and reflectometry, and magnetically by vibrating sample magnetometry and polarized neutron scattering. The experimental results are compared to the properties of the non-interacting nanoparticles in order to elucidate the impact of dipolar interaction in different structural hierarchies. Our approach and the new high quality model systems of layered nanoparticle arrays allow access to systematic studies of dipolar magnetic interactions of nanostructures across well-defined length scales.

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## Topologically stable helices in exchange-coupled rare-earth/rare-earth multilayer with superspin-glass-like ordering

ID 21

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The magnetization process of a two-dimensional randomly anisotropical system is directly connected with topologically stable helices in the form of 2 -planar domain walls (DWs). These DWs are often reported for rare-earth/transition metal layered systems. In this paper, we investigate such DWs within a rare-earth/rare-earth system, viz. [Dy/Tb]<sub>10</sub> multilayers with two different anisotropic layers. Superspin-glass (SSG) type of behavior was revealed by DC magnetization and AC susceptibility measurements as a function of temperature and frequency. Interestingly, magnetic investigations on these samples revealed an exchange bias field upto  $-0.88$  kOe which is commonly reported in rare-earth/transition-metal films, but not in rare-earth/rare-earth films. Using polarized neutron reflectometry (PNR) at the neutron reflectometer MARIA at FRM II in Germany, we find evidences of superimposed helical ferromagnetic magnetic configurations within both Dy and Tb. These 2 -DWs have lead to spin-frustrated interfaces. Off-specular neutron scattering indicates vertically correlated structure and fluctuations of the magnetization around the mean magnetization from vertically uncorrelated domains. Fundamentally, the helical ground states coexistent with superspin-glass-like ordering are topologically stable. In principle, such systems can therefore be exploited in all-spin-based technology.



## Proximity effects across oxide interfaces of superconductor-insulator-ferromagnet hybrid heterostructure

ID 24

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A case study of electron tunneling or charge-transfer-driven orbital ordering in superconductor (SC)-ferromagnet (FM) interfaces has been conducted in heteroepitaxial YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> (YBCO) / La<sub>0.67</sub>Sr<sub>0.33</sub>MnO<sub>3</sub> (LSMO) multilayers interleaved with and without an insulating SrTiO<sub>3</sub>(STO) layer between YBCO and LSMO. X-ray magnetic circular dichroism (XMCD) experiments at BESSY in Germany revealed anti-parallel alignment of Mn magnetic moments and induced Cu magnetic moments in an YBCO/LSMO multilayer. As compared to an isolated LSMO layer, the YBCO/LSMO multilayer displayed a (50%) weaker Mn magnetic signal, which is related to the usual proximity effect. It was a surprise that a similar proximity effect was also observed in a YBCO/STO/LSMO multilayer, however, the Mn signal was reduced by 20%. This reduced magnetic moment of Mn was further verified by depth sensitive polarized neutron reflectivity at the neutron reflectometer MARIA at FRM II in Germany. Electron energy loss spectroscopy experiment showed the evidence of Ti magnetic polarization at the interfaces of the YBCO/STO/LSMO multilayer. This crossover magnetization is due to a transfer of interface electrons that migrate from Ti(4+)– to Mn at the STO/LSMO interface and to Cu<sup>2+</sup> at the STO/YBCO interface, with hybridization via O 2p orbital. So charge-transfer driven orbital ordering is identified as the mechanism responsible for the observed proximity effect and Mn-Cu anti-parallel coupling in YBCO/STO/LSMO. This work provides an effective pathway in understanding the aspect of long range proximity effect and consequent orbital degeneracy parameter in magnetic coupling.

PROXIMITY EFFECTS ACROSS OXIDE-INTERFACES OF HYBRID SUPERCONDUCTOR-INSULATORFERROMAGNET HETEROSTRUCTURE: C. L. Prajapat, S. Singh, D. Bhattacharya, G. Ravikumar, S. Basu, S. Mattauch, J-G. Zheng, T. Aoki and Amitesh Paul  
Sci. Rep. 8, 3732 (2018).

## Structural and magnetic properties of cobalt iron disulfide nanocrystals

ID 27

**PAUL, Neelima**

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We report on synthesis and investigation of nanocrystalline cobalt-iron-pyrites with an emphasis on nanocrystal structure, morphology and magnetic behavior. The nanocrystals (NCs) were 5– 25 nm in diameter as characterized using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Local atomic structures of the samples were studied using absorption near edge structure (XANES) and extended absorption fine structure (EXAFS) at the Synchrotron Light Research Institute (SLRI), Nakhon Ratchasima in Thailand. With an increase in Fe fraction, X-ray diffraction and small-angle-X-ray scattering (SAXS) showed a systematic decrease in lattice constant, primary grain/ NC size (15 to 7 nm), and nanoparticle (NP) size (70 to 20 nm), respectively. The temperature dependence of the DC magnetization and AC susceptibility versus frequency revealed a number of magnetic phases in  $\text{Co}_x\text{Fe}_{1-x}\text{S}_2$ . Samples with  $x = 1$  and  $x = 0.875\text{--}0.625$  showed evidence of superspin glass (SSG) behavior with embedded ferromagnetic (FM) clusters of NPs. For  $x = 0.5$ , samples retained their mixed phases, but showed superparamagnetic (SPM) behavior with antiferromagnetic clusters suppressing magnetic dipolar interactions. Below  $x = 0.5$ , the pyrites show increasing paramagnetic character. We construct a phase diagram, which can be understood in terms of competition between the various dipolar, exchange, inter- and intracluster interactions. Our results suggest that NC size and shape can be tuned to engineer spin-polarized ferromagnetism of n-doped iron pyrite.

STRUCTURAL AND MAGNETIC PROPERTIES OF COBALT IRON DISULFIDE  $\text{Co}_x\text{Fe}_{1-x}\text{S}_2$  NANOCRYSTALS: H. Gabold, Z. Luan, N. Paul, M. Opel, P. Müller-Buschbaum, M. Law, Amitesh Paul. *Sci. Rep.* 8, 4835 (2018).

## Aging studies and influence of anode in LiFePO<sub>4</sub>-based cells with neutron diffraction

ID 31

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Aging in identically prepared 18650-type LFP/C cells, differing only in the choice of the carbon anode, was investigated by neutron diffraction. In all cells, not only the irreversible capacity losses due to formation but also the active lithium losses due to cycling could be quantified by neutron diffraction and were validated by electrochemical measurements. The LFP/MCMB cell, having mesocarbon microbeads as carbon anode, showed an excellent performance, suffering only a 8% relative capacity loss on 1C charge-discharge for 4780 cycles, and no loss due to calendric aging. The LFP/NC cell, having needle coke as carbon anode, suffers a higher irreversible capacity loss, probably due to formation of a more unstable SEI layer. It also shows calendric aging as well as a poorer cycling performance compared to LFP/MCMB, with a 23% relative capacity loss under similar cycling conditions. This loss is mainly due to active lithium loss and can be detected by neutron diffraction. With neutron diffraction we can rule out capacity loss due to structural degradation, partial loss of carbon or LiFePO<sub>4</sub> active material from electrode delamination or particle isolation. In LFP/NC cells, we were able to recover part of the lost capacity at 1C by a slower charging. However, in LFP/MCMB cells, we were not able to recover any of the lost capacity by this procedure. Thus, kinetics plays a more crucial role in LFP/NC than in LFP/MCMB cells. The comparative poor cycling performance of the LFP/NC cell is probably due to the higher tortuosity of the NC anode.

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## JCNS @ ILL: News and progress on IN12

ID 38

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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 years 2010 to 2012 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]. 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 108 n/sec/cm<sup>2</sup> around  $k_i = 2 \text{ \AA}^{-1}$ . In addition an extended wavelength range far into the warmish region (max.  $k_i = 5.1 \text{ \AA}^{-1}$ ) is now available. IN12 further exhibits vast capabilities for the use of an extended sample environment, including high magnetic fields up to 15T. Of course the instrument is equipped for the use of polarized neutrons, full polarization analysis (even together with high magnetic fields at the sample) and a cryopad set-up are available as standard options. We will also report on the progress of the multianalyser system IN12-UFO that is currently in a commissioning phase. Further, neutron measurements and results from user experiments will be shown that demonstrate the features and capabilities of this powerful three-axis spectrometer.

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## Time-resolved X-ray diagnostics of pulsed laser ablation in liquids (PLAL)

ID 40

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PLAL is a robust technique to produce nanoparticles of a broad choice of materials by ablation with pulsed lasers. When this process takes place inside a liquid, nanoparticles are caught in suspension for further use. Nevertheless the understanding and control of particle yield and morphology is far from being understood. The complexity of the process originates in the inherent span of timescales from laser-matter interaction on picoseconds up to aging time scales of hours and the interplay of different length scales. The latter involves the confinement of the nascent particles in a transient vapor bubble with a lifetime below milliseconds. We have analyzed this process with time-resolved methods spanning the nanosecond to second time span by scanning SAXS [1], WAXS, X-ray imaging [2,3], X-ray spectroscopy [3] and optical methods to resolve some of the fundamental structure-formation processes.

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## Grating-based phase-contrast microtomography at PETRA III

ID 42

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The Helmholtz-Zentrum Geesthacht, Germany, is operating the user experiments for microtomography at the beamlines P05 and P07 using synchrotron radiation produced in the storage ring PETRA III at DESY, Hamburg, Germany. Attenuation-contrast and phase-contrast techniques were established to provide an imaging tool for applications in biology, medical science and materials science. Here we will present the current status of the grating-based phase-contrast setup including the development of a 20 MPixel high speed CMOS camera together with the optimisation of the used grating setup. Selected examples of user applications will be given.



## Voltage control of magnetism in oxide heterostructures: Neutron & X-ray and electron microscopy investigation

ID 43

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Voltage control of magnetism (VCM) in oxide heterostructures, such as  $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3/\text{BaTiO}_3$  is of considerable interest due to the strong coupling between lattice, charge, spin and orbital degrees of freedom at interfaces, as well as for improving the functionality of future spintronic devices. In this study, the manipulation of magnetization via switching of ferroelectric polarization at interfaces as a function of electric field is investigated. We make use of a combination of advanced scattering (neutron and X-ray) methods, electron microscopy and spectroscopy (including off-axis electron holography and electron magnetic circular dichroism). Various mechanisms are involved in VCM, such as carrier modulation, strain effect, exchange coupling and orbital reconstruction. The role of each mechanism in the mediation of VCM in oxide heterostructures will be discussed.

## The Structural and magnetic properties of ordered arrangements of magnetic nanoparticles

ID 44

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Magnetic nanoparticles and their assembly in highly ordered structures are fundamentally interesting regarding the understanding of magnetic interactions and for a rational design towards potential applications in information technology as e.g. magnetic data storage media or as material for spintronics. With regard to these applications, the main aspects of fundamental interest include magnetic anisotropy, Van-der-Waals forces and interparticle interactions leading to aggregation or even ordered assemblies of nanoparticles. In order to obtain long-range ordering between magnetic nanoparticles in two and three-dimensions, we use pre-patterned substrates with a feature-size of the same order of magnitude as the diameter of the nanoparticles. A part of this work focuses on the structural and magnetic characterization of monolayers of CoFe<sub>2</sub>O<sub>4</sub> nanoparticles on silicon substrates. The system was characterized laterally by Scanning Electron Microscopy (SEM) and Grazing Incidence Small Angle X-ray Scattering (GISAXS) at the laboratory high brilliance GALAXI instrument [1] with simulation using the BornAgain software [2]. We deduce both the height profile of the individual nanoparticles, and a hexagonal ordering between the nanoparticles. Macroscopic magnetization measurements and polarized neutron reflectometry on a MARIA reflectometer [3] were used to find that the nanoparticles are weakly magnetized with respect to bulk CoFe<sub>2</sub>O<sub>4</sub> and that a random in plane relative orientation of the nanoparticle magnetizations is obtained at zero applied field. Also, we report on the structural characterization of pre-patterned silicon substrates. And the structural characterization of silica and magnetic nanoparticles that are self-assembled from toluenebased dispersions on flat and pre-patterned substrates laterally by Scanning Electron Microscopy (SEM) and vertically by Grazing Incidence Small Angle X-ray Scattering (GISAXS). Simulation and data analysis is performed using the Born Again software [2].

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## Morphology and crystallinity of $\text{Sr}_x\text{Co}_y\text{O}_z$ films at different growth conditions and stoichiometry

ID 46

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Because of its multivalent Co states and high oxygen mobility,  $\text{SrCoO}_{3-\delta}$  is a promising material for energy and information applications.  $\text{SrCoO}_3$  is a ferromagnetic metal with a Curie temperature of 305K, which becomes an antiferromagnetic insulator with a Néel temperature of 570K, when the oxygen content is decreased to  $\text{SrCoO}_{2.5}$ . Along with this magnetic transition, the structure changes from perovskite to brownmillerite, and the missing oxygen atoms form vacancy channels. We aim to grow thin films of  $\text{SrCoO}_{3-\delta}$  by molecular beam epitaxy and to fill the oxygen vacancies by annealing in a tube furnace under constant oxygen flow. These films are investigated by in-situ electron diffraction, ex-situ X-ray reflectometry and diffraction as well as atomic force microscopy. The stoichiometry is determined by Rutherford backscattering spectrometry. To investigate the intercalation of oxygen into the film in depth dependence, neutron reflectometry is performed to determine the change of the magnetic structure. We present the effect of growth conditions on the stoichiometry, crystallinity and morphology of  $\text{Sr}_x\text{Co}_y\text{O}_z$  films on  $\text{SrTiO}_3$  and requirements for  $\text{Sr}_1\text{Co}_1\text{O}_{2.5}$  films. Results of decreased oxygen vacancies by annealing in a furnace under oxygen flow and corresponding changes in the structure and magnetic properties are shown, as well as neutron reflectometry measurements to determine the depth dependent magnetic profile.

## High-pressure crystallographic studies in diamond anvil cells using neutrons at HEIDI

ID 47

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High-pressure single-crystal X-ray diffraction in diamond anvil cells (DAC) using laboratory and synchrotron facilities can now be performed on complex crystal structures that are twinned or modulated. However, there are hardly any single-crystal neutron diffraction studies in the DAC with complete structural refinements. Even at the most advanced neutron facilities, it is difficult to investigate crystals with volumes below 1 mm<sup>3</sup> due to low neutron fluxes. The requirement for large samples also hinders a joint use of X-ray and neutron single-crystals diffraction upon compression. The combination of both techniques is highly advantageous for detailed studies on crystalline compounds with magnetic (dis) order and/or on compounds containing light elements. Recently, we have started to explore the feasibility of neutron measurements in the DAC on the single-crystal diffractometer HEIDI at the Heinz Maier-Leibnitz Zentrum in Garching that offers the benefit of short wavelengths with high fluxes. Currently, the minimum crystal size that could be studied there in a DAC is about 0.1 mm<sup>3</sup>. We have developed optimized DACs for measurements at room and low temperatures in the transmission and radial (panoramic) neutron scattering geometries. Some of these DACs could well be used for combined X-ray and neutron investigations. The diffraction data collected at HEIDI can be used for full and reliable structure refinements.



## Frustration in Sm-based pyrochlores

ID 63

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Samarium based pyrochlores have two important peculiarities relative to other rare-earth pyrochlores systems: the very small ordered magnetic moment of the  $\text{Sm}^{3+}$  ion and the high neutron absorption of its natural isotopic abundance hinder a better characterization of its magnetic properties at low temperatures. Apart from it,  $\text{Sm}_2\text{Ti}_2\text{O}_7$  and  $\text{Sm}_2\text{Sn}_2\text{O}_7$  present many of the same magnetic behavior of their isomorphous sister compounds, which include the suppression of longrange magnetic order down to sub-Kelvin temperatures due to the geometrical frustration imposed by the pyrochlore lattice. In this work, we present bulk and neutron measurements performed on isotopic enriched samples of the aforementioned compounds. The field susceptibility versus temperature shows that both pyrochlores present a weak antiferromagnetic coupling with a small negative Curie-Weiss temperature. Magnetization curves versus field measured down to 2 K reveal that the crystal field ground state of the Sm ions can be regarded as a well isolated Kramers doublet with Ising single-ion anisotropy. Heat capacity of  $\text{Sm}_2\text{Ti}_2\text{O}_7$  and  $\text{Sm}_2\text{Sn}_2\text{O}_7$  present a sharp anomaly at 350 mK and 450 mK, respectively. This anomaly is shown to correspond to the onset of an all-in-all-out long-range order in the stannate sample.

## Combination of kinetic and structural studies of catalysts at the CAT-ACT X-ray spectroscopy beamline at KIT

ID 64

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The CAT experimental station at the CATalysis-ACTinide wiggler beamline [1] at the Karlsruhe Institute of Technology (KIT) focuses on synchrotron based characterization of catalysts under realistic reaction conditions, e.g. in the fields of exhaust gas after-treatment, selective oxidation, energy related applications and sustainable fine chemical processes. A dedicated infrastructure including stationary reactive gas supply, gas dosing units and on-line product analysis as well as in-situ and operando cells for sample environments close to industrial reactors (temperature up to 950°C, pressure up to 50 bar, liquid/gas phase, etc.) are essential aspects of CAT. The present X-ray absorption experiment at the CAT station can be combined with complementary characterization techniques like X-ray diffraction [2, 3] and infrared spectroscopy [4-6]. This allows complementing the analysis of the local molecular environment of catalytically active elements with information about long range order structures and adsorbed reaction species. The unique possibilities for in-situ and operando studies of catalysts available at the CAT station will be illustrated by exemplary studies on direct synthesis of hydrogen peroxide from hydrogen and oxygen as well as methanol, dimethyl ether and Fischer-Tropsch synthesis.

1. Zimina, A., et al., Rev Sci Instrum 88, 113113 (2017)
2. Grunwaldt, J.D., et al., J Catal 194, 452 (2000)
3. Clausen, B.S., H. Topsøe, and R. Frahm, in Advances in Catalysis, D.D. Eley, et al., Editors, Academic Press. p. 315-344 (1998)
4. Marinkovic, N.S., Q. Wang, and A.I. Frenkel, J Synchrotron Rad 18, 447 (2011)
5. Newton, M.A., et al., Chem Commun 21, 2382 (2004)
6. Gänzler, A.M., et al., J Phys Conf Ser. 712, 012045 (2016)

## Magnetic properties and lattice dynamics of Mn<sub>3</sub>Fe<sub>2</sub>Si<sub>3</sub> single crystal

ID 66

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FRIESE, Karen (Jülich Centre for Neutron Science-2/Peter Grünberg Institut-4, Forschungszentrum Jülich GmbH);  
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We have investigated the macroscopic magnetic properties of a magnetocaloric Mn<sub>3</sub>Fe<sub>2</sub>Si<sub>3</sub> single crystal, which indicate a strong anisotropy. Applying the field perpendicular to c-direction, the magnetic response exhibits two features at T<sub>1</sub>≈123 K and T<sub>2</sub>≈69 K, and only one feature in field parallel to c-direction at T<sub>2</sub>≈ 69 K. Even above T<sub>1</sub> the magnetic response is far from Curie-Weiss behavior, indicating the significance of short range magnetic correlations. To elucidate the spin and lattice dynamics in the system, neutron inelastic scattering was carried out on the MERLIN time-of-flight spectrometer [1]. Taking advantage of the repetition rate multiplication (RRM), five separate incident energies (9 meV, 13 meV, 21 meV, 40 and 108 meV) were recorded simultaneously to explore the excitation spectrum in a wide range with adapted resolution conditions. Besides that, the experiment, which provides a complete coverage of large portions of reciprocal space, clearly showed that the magnetic structure of this compound is more complex than previously thought.

[1] R. I. Bewley et al, Physica (Amsterdam) 385B–386B, 1029 (2006).

## KWS-1 SANS instrument with polarization analysis

ID 67

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The KWS-1 small-angle neutron scattering (SANS) instrument is 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 [1]. Among the available options the most important concerns the studies of magnetic samples for which the instrument is equipped with transmission supermirror polarizer, adiabatic radio-frequency spin flipper and a recently obtained dedicated magnet and polarization analyzer. The three-channel V-cavity polarizer with Fe/Si coated supermirrors ( $m=3.6$ ) has an average polarization  $> 93\%$  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 wavelengths. A custom designed hexapod allows heavy loads and precise sample positioning in beam (also for grazing incidence SANS under an applied magnetic field). For the experiments with the polarization analysis a  $^3\text{He}$  analyzer is utilized. Due to space limitations in the sample area a special highly shielded sample magnet was ordered, which allowed close positioning of the  $^3\text{He}$  cell to the magnet. The magnet has two orthogonal horizontal accesses. For the maximum field of 3 T (parallel to the beam) the decay time,  $T_1$ , of the  $^3\text{He}$  cell approximately 50 cm away from the center of the magnet constituted 90 hours. The maximum analyzed  $q$  is  $0.06 \text{ \AA}^{-1}$ . All instrument components are running under a flexible instrument control system (NICOS).

[1] A. Feoktystov, H. Frielinghaus, Z. Di, et al., J. Appl. Cryst., 48, 61 (2015).



## The New Small-Angle Neutron Scattering Instrument SANS-1 at MLZ – Features and First Results

ID 69

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We present the features of the instrument SANS-1 at MLZ, a joint project of Technische Universität München and Helmholtz Zentrum Geesthacht [1]. Measurements of the beam profile, divergence, flux and polarization are given for various positions along the instrument and agree well with simulations. SANS-1 features two interchangeable velocity selectors with 10% and 6%  $\Delta\lambda/\lambda$  and a TISANE 14-window double-disc chopper. This combination allows tuning flux, resolution, duty cycle and frame overlap, including time resolved measurements with repetition rates up to 10 kHz. A key feature is the large accessible Q-range facilitated by the sideways movement of the detector. Particular attention is paid to effects like tube shadowing, finite tube wall thickness and anisotropic solid angle corrections, that arise due to large scattering angles on an array of single  $^3\text{He}$  tubes, where a standard  $\cos^3$  solid angle correction is no longer valid. Dedicated to hard matter, materials science and magnetism, SANS-1 features a flexible, spacious sample area with a heavy duty goniometer and unique sample environment like a set of magnets, ovens and a bespoke dilatometer. We show some extreme examples and prospects for future experiments, e.g. to investigate the onset of crystallization in magnetic materials and a future high field magnet project. Finally we present the polarization analysis option that combines a new compensated MEOP and an integrated RF-flipper together with a 2.2 T magnet.

[1] S. Mühlbauer et al., NIMA 832, 297-305, (2016)

## Pseudo-Goldstone Magnons in the Frustrated $S=3/2$ Heisenberg Helimagnet $\text{ZnCr}_2\text{Se}_4$ with a Pyrochlore Magnetic Sublattice

ID 72

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Low-energy spin excitations in any long-range ordered magnetic system in the absence of magnetocrystalline anisotropy are gapless Goldstone modes emanating from the ordering wave vectors. In helimagnets, these modes hybridize into the so-called helimagnon excitations. Here we employ neutron spectroscopy supported by theoretical calculations to investigate the magnetic excitation spectrum of the isotropic Heisenberg helimagnet  $\text{ZnCr}_2\text{Se}_4$  with a cubic spinel structure, in which spin-3/2 magnetic  $\text{Cr}^{3+}$  ions are arranged in a geometrically frustrated pyrochlore sublattice. Apart from the conventional Goldstone mode emanating from the  $(0\ 0\ qh)$  ordering vector, low-energy magnetic excitations in the single-domain proper-screw spiral phase show soft helimagnon modes with a small energy gap of  $\sim 0.17$  meV, emerging from two orthogonal wave vectors  $(qh\ 0\ 0)$  and  $(0\ qh\ 0)$  where no magnetic Bragg peaks are present. We term them pseudo-Goldstone magnons, as they appear gapless within linear spin-wave theory and only acquire a finite gap due to higher-order quantum-fluctuation corrections. Our results are likely universal for a broad class of symmetric helimagnets, opening up a new way of studying weak magnon-magnon interactions with accessible spectroscopic methods.

## Morphologies and Solvent Distribution During Solvent Vapor Annealing of Block Copolymer Thin Films: In situ, Real-time GISAXS Investigations

ID 74

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Block copolymer (BCP) thin films have been proposed for a number of nanotechnology applications. Solvent vapor annealing (SVA) has emerged as a powerful technique for manipulating the structure of BCP thin films. Grazing-incidence small-angle X-ray scattering (GISAXS) allows studying the SVA process, providing detailed information of the pathways and mechanisms of SVA induced restructuring in BCP thin films [1]. We present a new method, namely SVA with two independently prepared vapors of different selectivities for the two blocks [2] at the example of a thin film from a cylinder-forming polystyrene-*b*-poly(dimethyl siloxane) (PS-*b*-PDMS) diblock copolymer. The film is first swollen in the vapor of *n*-heptane (strongly selective for PDMS), which is stepwise replaced by the vapor of toluene (weakly selective for PS). The initial cylindrical morphology is transformed into, among others, the lamellar one. To determine the distribution of the two solvents in the two types of nanodomains during SVA, we use the intensities of the Bragg reflections in the 2D GISAXS maps along with the measured swelling ratio of the film and relate these to the morphologies observed.

[1] D. Posselt, J. Zhang, D.-M. Smilgies, A. V. Berezkin, I. I. Potemkin, C.M. Papadakis, *Progr. Polym. Sci.* 66, 80-115 (2017).

[2] A. V. Berezkin, F. Jung, D. Posselt, D.-M. Smilgies, C. M. Papadakis, *Adv. Funct. Mater.* 1706226 (2018).

## In situ GISAXS Investigations of pH and Temperature Responsive Block Copolymer Thin Films during Swelling in Water Vapor

ID 77

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Responsive block copolymer thin films are of interest for many applications, e.g. as switchable membranes. Many of these systems are based on physical hydrogels, where a hydrophilic midblock is end-capped by hydrophobic end groups. Their dynamics can be tuned by employing temperature responsive polymers as end blocks. In thin films, a pH-responsive midblock may be used to tune the self-assembly process, while the end blocks may be immobilized by temperature variation and the so created nanostructure may be frozen. The latter feature may be exploited in solvent vapor annealing (SVA), which is a technique to improve long-range order and alter the morphology in thin films, but has the drawback that morphologies achieved in the swollen state are often difficult to preserve during solvent removal. In the present work, thin films from the pentablock terpolymer P(n-BuMA8-co-TEGMA8)-b-PDMAEMA50- b-PEG46-b-PD-MAEMA50-b-P(n-BuMA8-co-TEGMA8) are investigated in-situ during SVA using grazing-incidence small-angle X-ray scattering (GISAXS). The films were prepared via spin coating from aqueous solutions of different pH and were subsequently subjected to SVA using water vapor, with experiments being performed at various temperatures. The pH during film preparation is found to play a major role: Films prepared at high pH do not show structural features in the GISAXS maps, even after SVA with water. In contrast, films prepared at low pH feature a well-defined spherical morphology, which is enhanced after SVA.

## New developments on the SANS instrument D11 at the ILL

ID 78

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D11 is the archetype of a small-angle neutron scattering, the longest ever built (2 x 40m), and the first one equipped with a two-dimensional detector. It is in user operation since 1972 [1] and serves many fields of scientific research from magnetism over chemistry and biology to soft condensed matter, all benefitting from an extended q-range accessible and the high flux of the ILL reactor. D11 covers a real space window from the nm towards the  $\mu\text{m}$  [2]. The majority of research projects coming to D11 fall into the soft condensed matter domain. This contribution will present ongoing upgrades on D11, in particular the new multi-beam USANS setup, inspired by the SAMBA approach of R. Gähler [3]. This option should attract new users as it allows to extend further the q-range down to  $\sim 3 \cdot 10^{-5} \text{ \AA}^{-1}$  while preserving a significant flux and allowing to record 2D patterns within 1 min to 1 h, at short wavelength to limit multiple scattering effects. The change of setup is fast (15 min) to let users switch between SANS and USANS modes at will. The second part of this contribution deals with the large choice of sample-environments available for SANS at ILL, illustrated by recent results. In particular, a new in-situ DLS implementation and the new high-pressure cells for SANS will be highlighted. Furthermore, the new layout of D11 as proposed and accepted in the framework of ILL's Endurance 2 upgrade program will be presented, notably with its high area, high count-rate detector and improved optics.

[1] K. Ibel, J. Appl. Cryst. 9 (1976) 296.

[2] P. Lindner, R. Schweins, Neutron News 21 (2010) 15.

[3] R. Gähler, B. Fák, R. Golub, T. Hills, S. Klimko, J. Lal, S. Prokudaylo, C. Stadler, Advances in Neutron Scattering Instrumentation, Proceedings of SPIE 4785 (2002) 153 .

## Impact of interfacial solvent restructuring onto catalytic behavior by PDF

ID 79

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Noble metal nanoparticles (NP) play a major role in modern heterogeneous catalysis. Reaction conditions like temperature and pressure impact the catalytic activity and selectivity. [1] Although a solvent effect is commonly accepted as a decisive parameter in liquid-phase catalysis, its fundamental understanding on a molecular level is still missing. Pair distribution function (PDF) analysis based on high-energy X-ray scattering experiments can access the size of and the molecular ordering within solvation shells around colloidal NPs. [2] To correlate solvent-dependent catalytic activity with the interfacial solvent structure, we performed the selective hydrogenation of styrene to ethylbenzene under atmospheric hydrogen pressure in various organic solvents like tetrahydrofuran (THF) and toluene. Dodecanethiol stabilized palladium (Pd) NPs with an average diameter of 3 nm acted as catalyst. The difference-PDF signal of the THF dispersions reveals four restructured layers of THF molecules stretching 2 nm away from the NP surface. Corresponding catalytic studies (gas chromatography) allow a structure-activity correlation. Finally, GC-MS can be coupled to in-operando PDF measurements (1 min time resolution) to track structural changes of both NP and interfacial solvent layers in liquid-phase catalysis.

[1] U. K. Singh et al., Appl. Catal., A 2001, 213, 1

[2] Zobel, M. et al., Science 2015, 347, 292



## Hard X-ray spectroscopy of magnetic thin films for spintronic devices

ID 81

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*DRUBE, Wolfgang (Deutsches Elektronen-Synchrotron DESY, Hamburg); FECHER, Gerhard H. (Max Planck Institute for Chemical Physics of Solids, Dresden)*

High tunnel magneto-resistance is a characteristic of high quality magnetic tunnel junctions (MTJs) indicating a high spin polarization and epitaxial interfaces. We have studied the electronic properties of buried thin films promising as base electrodes for MTJs. In particular, the influence of the stoichiometry and annealing on the shape of the core levels and the valence band was investigated. The main method used is hard X-ray photoelectron spectroscopy where the excitation by hard X-rays in the range of typically 3-10 keV produces energetic photoelectrons which carry electronic structure information from well below the sample surface (10-30 nm) making it a powerful tool for studies of complex materials, buried nano-structures and multi-layered structures relevant for device applications. The experiments were carried out at PETRA beamline P09.

## Directional, hierarchical films via spray coating

ID 82

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The supply of clean, safe and renewable energy is an essential human task. For the benefit of this, it is of reasonable interest to enable energy consumers satisfying their own demand based on renewable energy sources. Towards this self-sufficient and “green” energy supply, the possibility of large scaled and cheap roll to roll solar cell fabrication based on organic materials is an important step. With the potential of flexible, semi-transparent properties these solar cells open new paths of design and application, for example integrated in architecture, clothing and all day accessories. Enhancement of these solar cell’s lifetime and efficiency is a key challenge, for which it is necessary to understand and control the morphology and formation of the solar cell layers during the deposition process. We focus on providing spray coating as a roll to roll compatibility technique in order to investigate directional, functional material deposition. We address metal-biopolymer composite films as a new class of materials, which has high potential in a green energy scenario. For example, protein nanofibrils offer a way of templated structuring and directed material synthesis. It is the aim to install hierarchical nanostructures by mixing metal and polymeric colloids. In situ GISAXS and GIWAXS measurements will display the complex morphology evolution during the spray coating process. The resulting directional optical and electrical properties will be characterized by methods such as ellipsometry and conductivity measurements.

## The anomalous breakdown of the Stokes-Einstein relation in Ge-Sb-Te and Ag-In-Sb-Te alloys and its connection to fast crystallization in the supercooled liquid

ID 83

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Phase-change materials such as Ge-Sb-Te and Ag-In-Sb-Te alloys can be reversibly switched between amorphous and crystalline states on a timescale of nanoseconds. These alloys possess unique features such as ultrafast switching, strong electrical/optical contrast, and a relatively stable amorphous state, which are interesting for non-volatile computer memory devices and neuromorphic computing applications. While significant progress has been made on the understanding of their crystalline phases and bonding natures, the liquid-state behavior (especially supercooled liquid state) has been less explored and its connection to crystallization kinetics needs to be better understood. Here we characterize the structural relaxation on the timescale of picoseconds and the self-diffusion coefficients of the Ge-Sb-Te and Ag-In-Sb-Te alloys in a high fluidity state above their melting points using quasi-elastic neutron scattering (QENS) on the time-of-flight (TOFTOF) spectroscopy. We find experimental evidence of an anomalous breakdown of the Stokes-Einstein relation in this high atomic mobility state. The breakdown shows the same features of the wellknown anomalous liquid water. We also show that the origin of this breakdown is unlikely the dynamic heterogeneities that have been usually discussed in the deeply undercooled viscous liquid. Rather it may be related to a liquid-liquid transition (and possibly a liquid-liquid critical point) hidden below the melting temperature in the supercooled liquid state. The liquid-liquid transition in supercooled phase-change materials is also a semiconductor-metal transition and a fragile-strong transition. The anomalous liquid behavior may explain why this class of material has ultrafast crystallization kinetics at high temperatures, favorable for fast data read/write speed in memory devices, while its amorphous phase retains relatively stable at room temperature for long-time data retention.

## Spin Structure in Magnetic Nanospheres

ID 84

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We will present a combined study of X-ray and neutron small-angle scattering (SAXS/SANS) and magnetization measurements, which resolves in detail the chemical and magnetic morphology of magnetic nanoparticles. In particular, polarized small-angle neutron scattering allows to resolve the spatial magnetization distribution inside of magnetic nanoparticles and to discriminate the spin disorder contribution at the particle surface and in the magnetic core[1]. Usually, the reduced magnetization in magnetic nanoparticles is attributed to the surface spin canting or formation of a magnetic deadlayer. Field-dependent polarized SANS experiments on the spherical magnetic nanoparticles under study result in a reduced and field-variable magnetized particle volume, which indicates significant deviations from single domain behavior with constant particle moment. Even at a high magnetic field of 1.2 T, spin disorder at the nanoparticles surface amounts to 11% of the particle volume. In-depth analysis shows that the overall reduced magnetization in spherical magnetic nanoparticles results not only from surface spin disorder, but also from reduced magnetization inside the magnetized core. HRTEM indicates a defected structure in the particle core, which may lead to a non-homogeneously magnetized spin structure.

[1] S. Disch et al., New J. Phys. 14 (2012) 013025

## A compact and calibratable von Hamos X-Ray Spectrometer based on two full-cylinder HAPG mosaic crystals for high-resolution XES and RIXS

ID 86

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In high-resolution X-ray Emission Spectroscopy (XES) crystal-based Wavelength-Dispersive Spectrometers (WDS) are being applied for characterization of nano- and microscaled materials. Thereby the so called von Hamos geometry provides high detection efficiency due to sagittal focusing using cylindrically bent crystals. To maximize the detection efficiency a full-cylinder optic can be applied. A novel calibratable von Hamos X-ray spectrometer based on up to two full-cylinder optics was developed at the PTB. To realize the full-cylinder geometry Highly Annealed Pyrolytic Graphite (HAPG) [1] was used. Besides its good bending properties this mosaic crystal shows highly integrated reflectivity while offering low mosaicity ensuring high resolving power [2]. The spectrometer enables chemical speciation of elements in an energy range from 2.4 keV up to 18 keV. Using synchrotron radiation as a tunable excitation source resonant inelastic X-ray scattering (RIXS) can be carried out with the spectrometer. The design and commissioning of the spectrometer will be presented together with first results using synchrotron radiation as excitation source. The spectrometer combines high efficiency with high spectral resolution (ten times better than in commercial WDS systems) in a compact arrangement also suitable for laboratory arrangements.

[1] H. Legall et al. (2006). Proc. FEL, BESSY FRAAU04, 798 – 801

[2] M. Gerlach et al. (2015). J. Appl. Cryst. 48, 2015, 1381-1390

## Structure investigation of the new VDM Ni-based superalloy 780 Premium

ID 87

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Ni-based superalloys are used for high temperature (T) applications that require good mechanical properties. Among these alloy 718 is the most widely used with operation T up to 650°C. In this alloy the austenitic matrix is strengthened by  $\gamma'$ -Ni<sub>3</sub>Al and  $\gamma''$ -Ni<sub>3</sub>Nb precipitates. Other phases that can be formed are  $\gamma'$ -Ni<sub>3</sub>Nb and  $\gamma''$ -Ni<sub>3</sub>Ti. The existence of the different phases, quantity and shape of the precipitates depend on composition, heat treatment and processing conditions. It is crucial to control their evolution at high T in order to tailor the mechanical properties. The aim of increasing the operation T forces the development of materials stable at higher T. Waspaloy has higher amount of  $\gamma'$  phase which allows its use at higher T but has a poor hot formability. The alloy 718Plus improves the performance at high T and it is expected easier processing. With improved composition new VDM alloy 780 Premium has been developed for higher service T and its structure and performance is under investigation. This work presents the in-situ structural characterization of the new VDM alloy 780 Premium after three different aging conditions by means of neutron and X-ray diffraction at room T and elevated T. The morphology of the different phases is studied by scanning electron microscopy and small angle neutron scattering. Furthermore, the solvus T of the different phases is determined and the structural properties correlated with hardening of the materials.



## Studying the dependency between ligand and Gold to improve nanoparticle growth

ID 90

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Gold nanoparticles (AuNP) are widely applied in many different scientific fields like medicine, biology and optics. Especially anisomorphic AuNP like rods (AuNR) are highly versatile and grown out of the liquid phase via a seed mediated growth process aided by a soft template consisting of ligands like CTAB. However, a major problem is the lack of control during the nanoparticle growth due to the highly complex interplay between the different reactants, additives and stabilizing agents. Our focus lies on the study of the soft template and its influence during growth on the AuNR morphology and yield. For that we use a combination of SAXS and SANS. With SAXS we can determine all properties concerning the AuNP. SANS on the other hand is in our experience a very reliable method to observe the soft template in its naturally occurring state under different conditions. We found, that despite the widely cited theory of a closed surfactant bilayer stabilizing AuNPs, the surfactant micelles play a major role during the AuNP growth. By tuning the morphology of micelles, via the addition of different additives, we could guide AuNR growth. We directly correlated an increased micelle length, with increased colloidal stability, in the case of the seed particles, and decreased aspect ratios for AuNR. Future work will expand on that research to find a consistent stabilization model, which includes the micelles as a major factor, to find new pathways for an improved control over the synthesis.

## Development of a cylindrical MiniMuPAD

ID 99

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Spherical neutron polarimetry was systematically applied over the last two decades. This technique allows to determine all nine components of the depolarisation matrix and to solve the Blume- Maleyev equations. It offers to investigate magneto-electric coupling, non-collinear magnetic structures, the chirality of magnetic scattering or to distinguish between spin flip and non-spin flip scattering. Therefore it allows for the separation of nuclear and magnetic scattering. Inspired by the cryogenic polarisation analysis device (CryoPAD) by Tasset in 1989, the Mu-metal Polarisation Analysis Device (MuPAD) presented by Janoschek et al, followed by a more compact form, namely MiniMuPAD, introduced by Haslbeck and Kindervater, we want to report on the currently being developed cylindrical MiniMuPAD. It is comparable to the existing MiniMuPAD, but now the precession coils are bent into cylindrical shape around the sample. It is, hence, no longer restricted to small angles as before. Further, due to its size, it is easy to handle and can be combined with a cryostat.

## Neutrons as tool for Residual Stress characterization from the surface to the bulk – RS state improvement of a 316L ITER welded plate by machining

ID 106

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Welding generally introduces unwanted local residual stress states on engineering components hindering a high quality performance in service. Most common procedures to reduce the undesired tensile residual stresses are post heat treatments or mechanical surface treatments as e.g. hammering or shot peening. However, all of these post treatment processes are elaborate and rather cost-intensive. The question arises if successive surface machining can be applied to effectively mitigate detrimental near surface tensile residual stresses. Within the framework of the Task Group (TG4) of the NeT project (The European Network on Neutron Techniques Standardization for Structural Integrity) a three pass slot weld made from austenitic stainless steel 316L has been manufactured with the aim to undertake 3-dimensional analyses of these residual stresses by both experimental and numerical means [1-2]. In this presentation we report on the effects of successive surface machining on the residual stress in a welded TG4 austenitic steel plate. The residual stress profile was determined experimentally using neutron diffraction measurements from the surface (100  $\mu\text{m}$ ) into the bulk and the results are compared and discussed with other experimental methods and finite element simulations – FEM. The numerical simulations use a dedicated “hybrid method”, specifically set up to simulate finish milling, which has been subsequently applied to the welding simulation so as to predict the final state in the component and its interaction with previous operations. The near surface neutron diffraction data were corrected for spurious strain effects due to the instrument setup and data smearing inside the gauge volume using an analytical approach developed for STRESS SPEC instrument [3-5].

1. M.C. Smith, A. Smith, R.C. Wimpory, C. Ohms, Proceedings of ASME 2015 Pressure Vessels and Piping Conference, Volume 6B: Materials and Fabrication, Boston, Massachusetts, USA, July 19-23, 2015, Conference Sponsors: Pressure Vessels and Piping Division
2. F. Valiorgue, A. Brosse, V. Robin, P. Gilles, J. Rech, J.M. Bergheau, Proceedings of ASME 2013 Pressure Vessels and Piping Conference, Volume 6A: Materials and Fabrication, Paris, France, July 14-18, 2013, Conference Sponsors: Pressure Vessels and Piping Division
3. J. Saroun, J. R. Kornmeier, M. Hofmann, P. Mikula, M. Vrana, J. Appl. Cryst. 46 (2013) 628–638
4. J. R. Kornmeier, J. Šaroun, J. Gibmeier, M. Hofmann, Mat. Sci. Forum 768 769 (2014) 52-59
5. J. Saroun, J. R. Kornmeier, J. Gibmeier, M. Hofmann, Physica B Condensed Matter, (2018)

## Grain boundary self-diffusion in $^{56}\text{Fe}/^{57}\text{Fe}$ multilayers by in situ neutron reflectometry

ID 109

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For the stability of nanostructured materials, grain boundaries (GBs) can be controlled by the grain size. When the diffusion length  $L_d = (2Dt)^{1/2}$  is small compared to  $\sim 0.5$  nm, the GB width, the volume diffusion at low temperature is slow compared to the GB diffusion (type-C regime),  $D$  being the volume diffusivity and  $t$  the isothermal annealing time. We studied self-diffusion active at the GBs at temperatures  $150^\circ$ ,  $175^\circ$ ,  $200^\circ$  and  $225^\circ$  C (consecutively for 600 min each) from the change of the  $^{57}\text{Fe}$  isotopic fraction monitored in situ by neutron reflectometry on the subnanometer length scale on  $\text{Pt}(4 \text{ nm})[^{56}\text{Fe}(x \text{ nm})/^{57}\text{Fe}(x \text{ nm})]_4/\text{Si}$  with ( $x=4$  and  $8$ ), in a dedicated furnace at REFSANS of HZG at FRM II in TOF mode. The samples of different thicknesses represent different average grain sizes without their evolution within a sample [1]. In situ experiments is expected to follow the dynamics more efficiently and therefore the mixed interface width has been mapped with time at short intervals. Specular profiles, extracted from the 2D detector maps, were analyzed by the Fitsuite code ([www.fs.kfki.hu](http://www.fs.kfki.hu)) simultaneously at each temperature as a function of momentum transfer vector  $Q$  and annealing time  $t$  to extract the GB diffusion coefficient. The regular diffusion (decaying Bragg peak) regime starts above  $200^\circ\text{C}$ . We find an increase in the intensity of the first Bragg peak at lower temperatures which is probably related to initial smoothening of the interfaces of the as-prepared state of the multilayer. We plan to analyze this aspect further from the corresponding off-specular scattering in the near future.

[1] J. Gong et al, RSC Adv. 7, 9573 (2017)

## Instrumentations for in situ PXRD and XAS during the adsorption of gases and vapours at KMC-2 beamline of BESSY II synchrotron

ID 114

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Metal-Organic Frameworks – a class of crystalline porous materials with high surface area and pore volume and application potential ranging from gas storage and catalysis to electronic devices. One of the unique features of MOFs is adsorption induced structural switching. In order to follow the phase transitions and pore filling mechanism during the gas and vapour adsorption, three different types of instrumentations were established at KMC-2 beamline of BESSY II synchrotron. First one is dedicated to monitoring of the structural changes in flexible MOFs during low-pressure (up to 1 bar) adsorption of non-corrosive gases at temperatures close to the boiling point of the fluid. The second type of the instrumentation provides a possibility to measure PXRD patterns in parallel to a high pressure adsorption (up to 80 bar). The main concept of the third type of the cell is to provide deeper insights into the mechanism of the pore filling during the adsorption of vapours, which is one of the critical points for selection of materials for application in adsorptive heat pumps. Low- and high-pressure gas cells are commissioned at KMC-2 beamline of BESSY II and are available for users of the large scale facility. The vapour cell is now under commissioning and will be available by middle 2019.

## Histidine protonation and its influence on the electronic and vibrational properties of a “Rieske-like” iron-sulfur protein

ID 117

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Iron-sulfur (Fe-S) clusters play a central role in energy transduction and electron transport. Unlike “ferredoxin-like” Fe-S clusters, Rieske centers contain a 2Fe2S cluster with one Fe coordinated by two histidines (FeN) and one Fe coordinated by two cysteines (FeS). A special feature of the Rieske centers is the pH dependence of their reduction potentials. Protonation of the N $\epsilon$ 2 atoms of the two imidazole rings coordinated to the Fe-S cluster is coupled with cluster reduction (electron transfer) in the *Thermus thermophilus* Rieske protein. Here, we present a nuclear forward scattering (NFS) and nuclear inelastic scattering (NIS) study on a “Rieske-like” Fe-S protein from *Saccharomyces cerevisiae* in order to investigate the influence of protonation on the electronic structure of the Fe-S cluster and on the Fe-ligand vibrations. NIS data sets of <sup>57</sup>Fe enriched “Rieske-like” Fe-S protein at three different pH values (6.4, 8.5 and 10.4) reveal pH dependent vibrational bands in an area where the FeN-histidine modes occur. In order to explain this effect and to get a deeper understanding of the coupling of the electron transfer to the protonation state of the coordinating histidines, NIS data have been simulated by means of combined quantum chemical and molecular mechanics (QM/MM) calculations based on a model for a 2Fe2S “Rieske-like” cluster with different His-ligand protonation states.



## Measurement of the Vacancy Formation Enthalpy of Lanthan at the CDB Spectrometer at NEPOMUC

ID 124

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The coincident Doppler broadening (CDB) spectrometer at the positron beam facility NEPOMUC of the research neutron source FRM II enables depth dependent and spatially resolved defect studies by using Doppler broadening spectroscopy (DBS) of the positron-electron annihilation line. In order to investigate the near-surface region and the bulk of a sample, the positron implantation energy can be set up to 30 keV. With a heatable sample holder temperature dependent in situ defect spectroscopy can be performed from room temperature up to 1000 K. Within this study the vacancy formation enthalpy of La and Cu as reference material was determined by temperature dependent DBS. First, the as-received samples were annealed in situ, i.e. the decrease of the so called S-parameter indicated the annealing of lattice defects. During a second heating cycle the increase of the vacancy concentration was clearly observed from which the vacancy formation enthalpy could be calculated.

## Cellulose-based conducting nanocomposite films via spray deposition with in situ GISAXS

ID 125

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In the emerging field of printable electronics there is a growing demand for transparent, flexible conductive materials. Cellulose-based substrates are a promising sustainable alternative to fully synthetic polymers. We present the fabrication of conducting composite films of cellulose nanofibers (CNF) and poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS). CNF of high surface charge are produced with TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl radical)-mediated oxidation and mixed with PEDOT:PSS in aqueous dispersion. Composite films are produced with a spray deposition followed in situ with GISAXS with sub-second time resolution. GIWAXS applied to the final films was used for the investigation of crystallinity in the composite. Different CNF/PEDOT:PSS ratios and the influence of the additive glycerol were investigated. The changes of the morphology and the influence on electric conductivity with the introduction of a compositional gradient are discussed. As the synchrotron-based investigation allowed for a high temporal resolution of 0.1 s, insights into the very first stages of the deposition process were obtained. The increase in conductivity can be explained by a change of morphology in the meso- and the nanoscale. The in situ investigation of the CNF/PEDOT:PSS composites are consistent with the application of glycerol as a plasticizer allowing for morphological changes especially with elevated temperatures as used with the spray deposition process.

## Element-specific atomic-scale structure and anion positions of $\text{Cu}_2(\text{Zn,Fe})\text{SnS}_4$ kesterite-stannite alloys

ID 128

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Substituting Zn by Fe in  $\text{Cu}_2\text{ZnSnS}_4$  changes the band gap of the material from about 1.5 eV to about 1.8 eV [Shibuya et al., Appl. Phys. Lett. 104, 021912, 2014]. Interestingly, the transition from  $\text{Cu}_2\text{ZnSnS}_4$  to  $\text{Cu}_2\text{FeSnS}_4$  is accompanied by a change of the crystal structure from kesterite type to stannite type via a complex rearrangement of the cation species [Schorr et al., Eur. J. Mineral. 19, 65, 2007]. Furthermore, the S anions in the mixed system are surrounded by different local cation configurations. In order to determine the element-specific bond lengths of the alloy material,  $\text{Cu}_2(\text{Zn,Fe})\text{SnS}_4$  powder samples with  $0 \leq \text{Fe}/(\text{Zn}+\text{Fe}) \leq 1$  were investigated with extended X-ray absorption fine structure spectroscopy. All bond lengths are nearly independent of the alloy composition, yet they differ substantially for the different elements. While the Cu-S and Fe-S bond lengths are identical, the Zn-S and Sn-S bond lengths are larger by about 0.03 and 0.12 Å, respectively. Based on these experimental results, the S anion position is modelled for different cation configurations and is found to be clearly different in the Zn or the Fe containing environment. This leads to an intrinsic structural inhomogeneity of the alloys on a subnanometer scale. The impact of the S anion displacement on the band gap energy was determined by density functional theory based calculations revealing a strong correlation between local atomic arrangements and electronic properties of  $\text{Cu}_2(\text{Zn,Fe})\text{SnS}_4$ .

## Continuous transition from antiferro- to ferromagnetic state via moment canting in $\text{Ni}_{2-x}\text{Co}_x\text{MnAl}$

ID 132

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The magnetic structure of matter, especially with regard to an application as functional materials, is often discussed only in terms of the classical concepts of ferromagnetism and antiferromagnetism. However, interesting phenomena can be expected when a system is driven to the boundary between these regimes by adjusting external parameters. The B2-ordered Heusler system  $\text{Ni}_{2-x}\text{Co}_x\text{MnAl}$  is a case in point:  $\text{Ni}_2\text{MnAl}$ , a potential ferromagnetic shape-memory material, displays antiferromagnetism [1], while  $\text{NiCoMnAl}$ , predicted to be a halfmetal, is ferromagnetic [2]. We have studied this system for compositions of  $0 \leq x \leq 0.8$  by temperature-dependent neutron powder diffraction at SPODI at the Heinz Maier-Leibnitz Zentrum (MLZ) Garching, observing antiferromagnetic ordering on the Ni-rich side that continuously decreases and vanishes at  $x = 0.4$ , while additional macroscopic magnetization measurements shows an increasing longitudinal component for all  $x > 0$ . We argue that this constitutes a continuous, spatially homogeneous transition from antiferro- to ferromagnetism via canted spins, resulting from the competition of the ferromagnetic Ni/Co-Mn interaction and the antiferromagnetic Mn-Mn interaction on the disordered Mn/Al sublattice. We reproduce our findings by a simple Heisenberg model.

[1] M. Acet et al., J. Appl. Phys. 92, 3867 (2002)

[2] P. Neibecker et al., Phys. Rev. B 96, 165131 (2017)

## Swift heavy ion-irradiated calcite (CaCO<sub>3</sub>) analyzed by UV-C Laser excited Fluorescence-Spectrometry

ID 135

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The mineral calcite (CaCO<sub>3</sub>, rhombohedral cell, 3 2/m symmetry, large band gap) is known to exhibit luminescence caused by electron excitation (cathodoluminescence), accelerated ion excitation (ionoluminescence), UV-light, and laser excitation. In most cases, the resulting wavelength can be traced back to impurities in the crystal lattice that act as luminescence centres. These impurities could be Pb<sup>2+</sup>, Mn<sup>2+</sup>, Ce<sup>3+</sup>, Sm<sup>3+</sup>, Nd<sup>3+</sup>, Dy<sup>3+</sup>, Eu<sup>3+</sup>, or radiation induced CO<sub>3</sub><sup>2-</sup>. The influence of natural radiation on luminescence behaviour of calcite leads to the assumption that it might be possible to determine the defect concentration, and therefore, the fluence applied to irradiated calcite crystals, by measuring the intensity change of the luminescence peaks. This could be a promising, non-destructive analytical technique to quantify defect concentrations that are caused by heavy ion-irradiation in condensed matter, in general. This poster addresses the relationship of artificially generated defect concentration by swift heavy ion irradiation and UV-C laser excited luminescence spectra of the mineral calcite. A new mobile UV-C laser excited fluorescence spectrometer system was built to be used at different irradiation beamlines (M-3 branch, SIS-18, and CRYRING) at GSI, Darmstadt, for online and in-situ measurements. The system consists of a Crylas 266-200 UV-C pulsed laser (of  $\lambda=266$  nm, 160  $\mu$ J/pulse, 60 Hz), a beam splitter, newly designed sample holder on a software driven 3-axis piezo-stage (PI Q521-300), a mirror, a UV-C beam dump, a longpass filter, two different optical fibres and two UV/Vis spectrometer. The Ocean Optics USB 4000 UV/Vis Spectrometer is used if the material under investigation provides high photon release. The Horiba Jobin Yvon iHR 320 spectrometer with a Pelletier cooled camera is used for low photon counts as it has a very high signal to noise ratio. Calcite crystals irradiated with 11.1 MeV/u Au ions of fluences between  $1 \times 10^6$  and  $1 \times 10^{12}$  ions/cm<sup>2</sup> were investigated with the new system. In comparison to non-irradiated calcite crystals, the following changes can be seen with increasing fluence:

- Increasing intensity of peaks and the appearance of new peaks.
- Non-irradiated: Intensity increase in the region of 500 to 600 nm for irradiated calcite (108 ions/cm<sup>2</sup>) compared to non-irradiated samples.
- For fluences between 108 and 10<sup>12</sup> ions/cm<sup>2</sup> intensity increase in the region of 550 to 700 nm.

The presentation will provide insight into the new analytical set-up and the first data on calcite irradiated with swift heavy ions.



## Application of X-Ray Fluorescence Holography on the Superconductor Parent $\beta$ -FeTe

ID 138

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In the structure of the superconductor parent  $\beta$ -FeTe (P4/nmm, #129) excess iron atoms are necessary to stabilize the system, leading to the formula  $\text{Fe}_{1+y}\text{Te}$ . This structure is built up of iron and Te layers. Diffraction experiments indicate for the excess Fe atoms partial occupation of interstitial positions in the Te plane, resulting in octahedral coordination. With the new technique of X-ray fluorescence holography (XFH) the atomic image can be recorded directly as the interference pattern of the Fe  $K\alpha$  fluorescence. With applying this method on  $\beta$ -FeTe we try to determine the position of the excess iron atoms without the usage of a structural model. Therefore, we have measured holograms of  $\beta$ -FeTe at two energies directly on the Fe K edge at the ESRF. Using the slight shift of the exact edge energy between the neutral excess Fe atoms and the charged Fe atoms of the regular Fe grid, we want to enhance the fluorescence emission of the two different Fe atoms selectively. From the holograms atomic images can be reconstructed using a sparse modeling algorithm.

## The structural, thermal and dynamic behavior of the thermoresponsive polymer poly(N-isopropylmethacrylamide)

ID 142

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Poly(N-isopropylmethacrylamide) (PNIPMAM) is a thermoresponsive polymer, exhibiting lower critical solution temperature (LCST) behavior in aqueous solution. Compared to the well-investigated poly(N-isopropylacrylamide) (PNIPAM), which has similar chemical structure, PNIPMAM has a higher transition temperature (43 °C [1] instead of 32 °C). This may be due to the presence of the additional methyl groups on the vinyl backbone, which lead to steric hindrance and weaken the intramolecular interactions [2,3]. In order to understand how these effects along with the hydrophobic interactions influence the phase behavior of aqueous solutions of PNIPMAM, we investigate the temperature-dependent phase behavior of PNIPMAM in D2O using turbidimetry, differential scanning calorimetry (DSC), small-angle neutron scattering (SANS) and dynamic light scattering, covering a large concentration range. We find that the phase transition consists of two steps, namely (i) hydrophobic aggregation at ~39 °C, as evident from turbidimetry, DLS and SANS and (ii) the chain collapse and water release at ~44 °C, as evident from DSC and SANS.

[1] K. Kunota et al., Polym. J. 22, 1051 (1990).

[2] J. Pang et al., J. Theor. Comput. Chem. 10, 359 (2011).

[3] J. Dybal et al., Vibrat. Spectrosc. 51, 44 (2009).



## Ordering of Carbon Tetrachloride confined in slit geometry

ID 144

**LIPPMANN, Milena**

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The structure of carbon tetrachloride (CCL<sub>4</sub>) created by confinement in slit geometry with gap size in the range of few tens of Angstrom is probed by reflectivity and in-plane x-ray measurement. The measurements reveal heterogeneous structures with regions ordered only in z-direction (layeredstructure) and periodic order in the plane of the substrates. Current experiments confirm the ability of confinement to induce crystal objects, which was long discussed in literature. The data analysis shows that in the case of CCl<sub>4</sub>, where the molecule has almost round shape, the spacing of the layered-structure is not close to the molecular size but to the atomic distances in the molecule. The confined films with thickness of 30-40 Å are in strong confined mode and the size of the layered system is smaller than correlation length in bulk. The experimental results suggest that the layered-structure in confinement resemble the short range molecular arrangement as observed in the bulk liquid with differences given by closer packing and modified mutual molecular orientation in confinement.

## Study of uranium in natural and synthetic carbonate apatites using radiochemical, synchrotron radiation and fission track techniques

ID 148

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Carbonate apatites were found to be present in U-bearing organic-rich phosphatized limestones from Epirus region (NW Greece). The mineralogical investigation of the rock samples revealed, along with organic matter and calcite, the presence of abundant carbonate apatites and in lesser extent fluoroapatites, zircons and pyrite. Electron microprobe (EMPA) and Raman spectroscopy were applied for the characterization of carbonate apatite crystals. Synchrotron radiation micro-X-ray fluorescence (SR  $\mu$ -XRF) measurements performed using as targets carbonate apatites demonstrated the uranium distribution, while micro-X-ray Absorption Near-Edge Structure ( $\mu$ -XANES) illustrated that U was present in the crystals tetravalent form (U(IV)). Fission track dating was attempted using both carbonate apatite and fluoroapatite crystals. Fluoroapatites were successfully etched and dated showing various ages. On the other hand, the dating of carbonate apatites was not successful because of the lack of a validated etching procedure. However, the high density of homogeneously distributed fission tracks in mica detector was an evidence of their high U content. The mechanisms of U adsorption onto carbonate apatites synthesized in the laboratory were studied and showed that this mineral could act as a significant U sorbent. Sorption experiments performed in solutions of pH 4 to 10 showed a partial dissolution of the synthetic carbonate apatite crystals surface and precipitation of meta-autunite ( $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 6-8\text{H}_2\text{O}$ ). The XRD patterns indicated that the formation of meta-autunite gradually decreased with increasing solution pH. Under very alkaline conditions (pH 13) meta-autunite did not appear. Regardless of this the carbonate apatites showed a high uranium uptake. In the latter case the unit cell of carbonate apatite crystals decreased indicating a possible substitution of Ca by U in the crystal lattice. In the current study along with a first approach to their etching method, new insights concerning the mineral chemistry of uranium in carbonate apatites are presented.

## PIPE: The Photon-Ion-Endstation at PETRA III for Experimental Studies of XUV-Photoprocesses in Small Quantum Systems

ID 152

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This contribution features the user facility PIPE which is a permanent end station at the Variable Polarization XUV beamline P04 at PETRA III / DESY for the study of photon interactions with matter in the gas phase (charged and neutral atoms, molecules, clusters). The setup provides flexible state-of-the-art experimental equipment such as specialized ion sources, an ion trap, and particle spectrometers with multiple coincidence capabilities. Since the start of operation in 2013, already several research highlights have been published, e.g., the quantitative study of a genuine four-electron process induced by resonant inner shell excitation of an atomic ion [Phys. Rev. Lett. 114, 013002 (2015)], the observation of a two-particle interference of electron pairs in a molecule [Phys. Rev. Lett. 117, 083002 (2016)], the imaging of the square of a correlated two-electron wave function in a molecule [Nature Communications 8, 2266 (2017)], or the determination of the probability for direct double ionization of an atomic anion by a single photon over a wide photon energy range [Phys. Rev. Lett. 120, 133202 (2018)]. The construction and building of the PIPE setup has been made possible by substantial funding from the German Ministry for Education and Research (BMBF) within the “Verbundforschung” funding scheme under contracts 05KS7GU2, 05KS7KE1, 05KS7RF2, 05KS7RG1, 05K10GUB, 05K10KEA, 5K10RF2, 05K10RG1, 05K13GUA, 05K16GUC, 05K16RF3, 05K16RG1.

## Strain Induced Martensitic Transformation in Austempered Ductile Iron (ADI)

ID 158

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Austempered ductile iron (ADI) is a nodular ductile iron which has undergone a special heat treatment to greatly enhance mechanical properties. This heat treatment process consists of austenitization, quenching to a temperature typically between 250°C and 450°C and isothermal austempering. Afterwards the microstructure consists of acicular ferrite and high carbon enriched retained austenite. The high carbon enriched retained austenite can transform to martensite during plastic deformation. Austempering temperature and time as well as alloying composition control the retained austenite fraction, grain size and its stabilization, which in turn will influence the following martensitic transformation. How different treatment and composition parameters influence the martensitic transformation has been investigated using in-situ neutron diffraction while applying either tension or compression to different plastic strains. In addition texture measurements using neutron diffraction have been performed to calculate the texture distribution of ferrite and austenite phases for different strain levels. Combining the detailed information on texture with the in-situ studies is necessary for quantitative phase analysis and extraction of martensite phase fractions. Here we will present the results from these experiments which allowed us to develop a model of the martensitic transformation kinetic in ADI due to plastic strains with respect to austempering temperature and alloying element content.

## Ultrafast neutralization dynamics of highly charged ions upon impact on 2D materials

ID 159

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The impact of ions on atoms, molecules and solid surfaces involves many different processes, among them charge exchange, electron excitation and ionization of the target, and subsequent deexcitation of both projectile and target. Experiments with slow ( $v < 1\text{a.u.}$ ) highly charged ions ( $q < Z$ ,  $Z \gg 1$ ) scattered off solid surfaces or transmitted through thin solid films revealed already some years ago the presence of a fast deexcitation process of the projectiles. This fast deexcitation was described by a rapid Auger-type cascade in the projectile proceeding in parallel combined with a continuous feeding of electrons from the target. By using a freestanding layer of graphene as the target and normal ion incidence with keV-100keV energies, we limit the interaction time of our ions with the material to some femto-seconds only, thus excluding any additional electron feeding from the target to the projectile during its deexcitation. Nevertheless, we observe a similarly fast de-excitation and neutralization of highly charged Ar and Xe ions within a time frame of  $< 10\text{fs}$ . Here we present a model of resonant electron capture in front of the surface according to the classical-over-the-barrier model and an interatomic deexcitation process with electrons of the surrounding target atoms (Interatomic Coulombic Decay (ICD)). We can now put observed charge exchange, energy loss and electron emission of slow highly charged ions impacting on solid surfaces on a common footing.

## Microstructure and in-situ tensile behavior of CNTs reinforced Mg composites using neutron diffraction

ID 160

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With extremely high elastic modulus, super strength, outstanding thermal and electrical properties, Carbon nanotubes (CNTs) are considered as one of the most potential reinforcements for composites. Our studies indicate that hybrid Mg MMCs, reinforced with SiCp and CNTs, have shown superior tensile properties. This is mainly attributed to the addition of CNTs to SiCp with CVD method. In this contribution, we will present first a brief introduction on a novel fabrication process of the CNTs reinforced Mg-Zn matrix composites, and then the characterizations of their microstructures and phase by lab X-ray. In-situ tensile deformation test of these composites was performed using neutron diffraction at STRESS-SPEC (MLZ, Garching). Peak position variation with the tensile strain of each phase was analyzed. Bulk texture of both the initial and the tensile to broken samples was also investigated ex-situ by neutron diffraction. X-ray results showed that in both as-cast and as-extruded materials there exists a precipitate of MgZn<sub>2</sub> which are formed during both casting and extrusion processes. The MgZn<sub>2</sub> was the only precipitate in ZK60 alloy and composites. The measured pole figures indicated no obvious change of the samples with the addition of CNTs. Lattice strain evolution via in-situ test indicated SiCp and CNTs reinforcement played a role in carrying internal stress during tensile deformation, and lattice strain showed shaper increase in CNTs reinforced composite than that in SiCp composite and ZK60 alloy, indicating CNTs bear the forces in whole tensile process.

## In-situ neutron diffraction study on compressive behavior of solution heat-treated Mg-Ca alloys at room and elevated temperature

ID 161

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The addition of Ca to Mg can improve the creep resistance at elevated temperatures and the mechanical properties of Mg alloys at both room and high temperatures. In current study, the in-situ compressive deformation behaviors of solution treated Mg-0.5Zr and Mg-0.3Ca-0.5Zr alloy were investigated both at room and elevated temperature (~200°C) at STRESS-SPEC neutron diffractometer (MLZ, Garching). Cylindrical samples with 6 mm in diameter and 11 mm in length were compressed using a unique tensile/compression rig at STRESS-SPEC. Peak position and intensity variation with the compression strain were analyzed since they can indicate the evolution of lattice strain and preferred orientation, respectively. This will be related to the effect of Ca addition on the deformation modes of Mg alloys including basal slip, {10.2} twinning, prismatic slip and pyramidal slip. Results showed that the addition of Ca strengthened all the deformation modes at room and elevated temperature. However, the hardening effect of Ca on prismatic slip was more significant than that on the other deformation modes at room temperature. All the deformation modes were softened at 200 °C, where prismatic slip and slip got easier to be activated and basal slip carried more internal strain than that as room temperature. The alloy with Ca addition showed a better thermal stability at 200 °C due to the significantly strengthening effect of Ca solutes.

## In situ high energy X-ray diffraction during transient liquid phase bonding of a $\gamma$ -TiAl alloy

ID 171

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TiAl alloys are increasingly being used as a lightweight material, for example in aero engines, which also leads to the requirement for suitable repair techniques. Transient liquid phase bonding is a promising method for the closure of cracks (in non-critical or non-highly loaded areas) and is already used for Ni-base superalloys. Two different brazing solders, based on Ti-Fe and Ti-Ni, have been investigated for brazing a  $\gamma$ -TiAl alloy. After brazing, the joints exhibit different microstructures and phase compositions. Additionally, tensile tests at room temperature show different mechanical strengths depending on the brazing solder. For a better understanding of the brazing process, the phases, their development and distribution over the brazing zone were investigated, time and position resolved, during the brazing process. These investigations were performed using high-energy X-ray diffraction at the HZG-run highenergy materials science beamline HEMS, located at the synchrotron radiation facility at DESY in Hamburg, Germany. During the analysis of the diffraction data with Rietveld refinement, the amount of liquid could be quantified using Gaussian peaks. Additionally, the degree of ordering of the  $\beta_0$  phase was determined with a model of two ideal stoichiometric phases (completely ordered and disordered). During brazing different phases occurred in the joint region over time. The phase composition changed clearly over the first six hours of the brazing process.

## An insight into atmospheric degradation processes by mapping local nanoheterogeneities within hybrid polycrystalline perovskite films

ID 174

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Hybrid perovskites are an exciting and a highly versatile class of materials within the photovoltaic community. Their versatility emerges from their crystallographic structure which allows for functionalization of materials into hybrid species by partial/ complete compositional substitution at various lattice sites. Their manufacturing is economically attractive since perovskite materials have low activation energies for crystallization and they require low temperatures for formation. However, as a result they are known to be sensitive to structural and chemical degradation in ambient atmosphere, especially in the presence of humidity and oxygen. Within this study, we map local crystallographic characteristics of two exemplary perovskite systems: a stoichiometrically pure and a mixed hybrid perovskite thin film. The samples were etched with a focussed ion beam and probed with the aid of a nanofocussed x-ray beam. Local heterogeneities emerging at the nanoscale were discovered, making it possible for the first time to map individual crystallites of solution-processed thin films and pinpoint local sites of degradation within the two prototypical materials.



## Morphology phase diagram of printed titania films derived from block copolymer template assisted a sol-gel technique

ID 177

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Printing, a simple and low-cost technique for producing films on an industrial scale, is employed to fabricate mesoporous titania thin films. The synthesis is performed by the combination of sol-gel chemistry, in which titanium(IV)isopropoxide (TTIP) is used as precursor and a diblock copolymer polystyrene-block-polyethylene oxide (PS-b-PEO) as the structure-directing template. The amphiphilic block copolymer PS-b-PEO undergoes phase separation and self-assembly due to a good-bad pair solvent, namely 1,4-dioxane and hydrochloric acid (HCl). By adjusting the weight fraction of 1,4-dioxane, HCl and TTIP, the titania films with different morphologies, containing foam-like structure, nanowire aggregates, collapsed vesicles and nanogranules, are obtained after evaporation and calcination processes. The surface morphology of the titania film is probed via scanning electron microscopy (SEM) and optical microscopy (OM), and the inner morphology is detected by grazing incidence small-angle X-ray scattering (GISAXS). The high crystallinity of anatase titania is proved by X-ray diffraction (XRD) and transmission electron microscopy (TEM) upon calcining nano-composite film at 450 oC in air.

## The Frag4Lead 3D Pharmacophore-Diverse Fragment Library and Following Up on Crystallographic Fragment Hits

ID 178

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The key idea of fragment screening is that a small selection of appropriate fragments covers a much larger proportion of the overall chemical fragment space than a typical high-throughput screening collection with respect to the drug-sized chemical space. Fragments may bypass strict steric requirements for binding, leading to high hit rates. They often efficiently bind in anchor positions making them excellent starting points for ligand design. However, to make the most out of a fragment screen it should adequately cover chemical space allowing to easily follow up on hits by available compounds and feasible chemistry. Our Frag2Xtal fragment library was designed to cover 3D-diverse yet representative pharmacophores intended for crystallographic screening against generic targets. This representativity allows easily following up on hits by available analogues and superstructures or by synthesis started from reactive synthons. We present a clustering- and docking-based strategy to select tailored fragment subsets and promising follow-up compounds from available candidates. Application to natural compound-derived fragment hits from AnalytiCon's FRGx library resulted in multiple superstructure ligands with improved affinity. The presented libraries and methods are part of the Frag2Xtal and Frag4Lead service facility for crystallographic fragment screening currently made available at the automated crystallographic BL14.2 at the BESSY II storage ring of the Helmholtz-Zentrum Berlin.

## Nuclear resonance scattering and Mössbauer studies of a trinuclear iron ferrocene complex

ID 181

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In order to study the cooperativity between the three irons of the trinuclear iron ferrocene complex  $[\text{Fe}\{\text{L-N}_4(\text{CH}_2\text{Fc})_2\}(\text{Cl})_2]$  synchrotron based spectroscopy such as nuclear inelastic scattering (NIS) and nuclear forward scattering (NFS) have been performed. These techniques are well suited to investigate valence tautomerism which denotes an intramolecular redox reaction over an energy barrier between two metal ions or between one metal ion and a redox active ligand. The NFS data display a quantum beat structure reflecting a quadrupole splitting of  $\Delta E_Q = 0.99 (\pm 0.02)$  mms<sup>-1</sup>. This is indicative of a ferric high-spin state for the central iron of this complex. The partial density of vibrational states (pDOS) determined experimentally by NIS measurements provides information about the vibronic properties of the complex. For a better understanding of these results, density functional theory (DFT) calculations have been performed on this complex. Furthermore, we have also carried out conventional Mössbauer spectroscopy to observe a possible electron transfer from the iron of the ferrocene ligand to the central iron ion. In addition, the zero field splitting D and the rhombicity E/D have been determined by measurements at 4.2 K and different external magnetic fields. This work is supported by the German Science foundation (DFG) via the SFB-TRR-88 3MET.

## Self-Assembly in ultrahigh molecular weight diblock copolymer thin films

ID 182

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Self-assembly of ultrahigh molecular weight diblock copolymer nanostructures with micro-phase separation structures larger than 150 nm can yield ordered structures for the fabrication of many nanotechnology materials, such as optical, electronic and magnetic devices. Therefore, it is significant to study the structural development in ultrahigh molecular weight diblock copolymer thin films systematically. In this work, the authors fabricate homogeneous polystyrene-block-polymethylmethacrylate diblock copolymer ( $M_n = 1061.60$  kg/mol) thin films, having a characteristic structure larger than 160 nm, with nitrogen gas ( $N_2$ ) to blow away the solvent vapor (dimethylformamid) inside the chamber for accelerating solvent volatilization during spin coating combined with a solvent annealing process. As a function of film thickness, we demonstrate a similar orientation evolution of cylindrical structures as known for normal molecular weight diblock copolymers. Well-ordered nanostructures with a vertical cylindrical morphology, average domain diameter of 82 nm and distance of 162 nm, are readily generated through the control of solvent annealing time. The film morphology is probed with optical microscopy (OM), atomic force microscopy (AFM), and grazing incidence small angle X-ray scattering (GISAXS).

## Preparation and characterization of spin crossover thin solid films

ID 183

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Iron(II) spin crossover (SCO) complexes indicate a reversible transition from the low-spin state (LS,  $S=0$ ) to a high-spin state (HS,  $S=2$ ) by e.g. variation of temperature, pressure or by irradiation with light [1]. Therefore, these materials are promising candidates for information storage [2]. However, practical device applications require thin films with these properties. The SCO-compound  $[\text{Fe}(\text{Htrz})_2(\text{trz})](\text{BF}_4)$  (1) switches between the LS and the HS state with a 30 K wide thermal hysteresis loop above room temperature [3]. We prepared thin films of 1 on a  $\text{SiO}_2$  substrate by spin coating. The spin states of the films were characterized by Mössbauer spectroscopy in reflection mode using a MIMOS II spectrometer. A low quadrupole splitting (LS state) at 300 K and a high quadrupole splitting (HS state) at 400 K were found for the films, as well as for bulk powder of 1. This confirms a LS – HS transition above room temperature. Furthermore, synchrotron based nuclear resonance scattering measurements from 80 K to 400 K indicate that the hyperfine parameters as well as the Fe-vibrational density of states of the film are similar to those of the bulk powder of 1. These studies show that the SCO films prepared by spin coating have similar SCO properties as the bulk complex.

[1] P. Güttlich, et al., *Angew. Chem.* 106, (1994), 2109-2141

[2] J. Kröber, et al., *Chem. Mater.* 6, (1994), 1404-1412

[3] F. Guillaume, et al., *Chemical Physics Letters* 604, (2014), 105-109

## Crystallographic screening of sp<sup>3</sup>-rich fragment library for Protein Kinase A

ID 184

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Over the last two decades, fragment-based lead discovery has resulted in two approved drugs and to more than 30 drugs in clinical trials<sup>1</sup>. One of the strengths of the fragment-based approach is the efficient sampling of the chemical space using small organic molecules with MW usually below 250 Da. Established screening libraries have been designed by combinatorial chemistry. The resulting profile of such libraries is dominated by sp<sup>2</sup>-carbons and aromatic rings, which undermines the chemical topology and molecular complexity of therapeutic drugs. Systematic studies found an increase of the sp<sup>3</sup>-carbon fraction, e. g. saturated rings, as the compound progresses from hit to the status of a drug candidate in clinical phases<sup>2</sup>. The deferral of molecular complexity and difficult chemistry of sp<sup>3</sup>-carbons to the later stages of drug development may also defer the risk of failure to costly clinical trials. In the present study, we utilized CrystalsFirst's SmartSoak® technology and performed a crystallographic screen of 200 fragments derived from natural products comprising a high fraction of sp<sup>3</sup>-carbons. The preliminary results show an extraordinary hit rate over 30 % that allows a wide spectrum of follow-up compounds using analogues-by-catalogues approach or by classic organic chemistry.

(1) Erlanson et al., Nat. Rev. Drug Discov. 2016, 15 (9), 605–619.

(2) Lovering et al., J. Med. Chem. 2009, 52 (21), 6752–6756.

## Initial investigations of hydrating cellulose thin films using GISANS

ID 186

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Due to its nanoscale architecture, cellulose nanofibrils (CNF) are used in functional materials as building blocks for high-strength materials [1], barrier coatings and functional coatings in thin films for organic electronic applications. One crucial question is the relation between packing of the CNF, porosity and nanostructure in the range from some nm (fibril diameter) to several hundred nm (fibril length) inside the thin film. The CNF might be of globular morphology or more elongated, depending on the film thickness and surface charge. The knowledge of the nature of porosity of the thin film is crucial for subsequently functionalizing the pores by filling with functional polymers. In order to approach a determination of the CNF nanostructure inside the thin film, we performed grazing incidence small angle neutron scattering (GISANS) at KWS-1 (JCNS, Garching). The thin film was prepared by airbrush spray coating [2]. We used TEMPO CNF with a surface charge of 1000  $\mu\text{mol/g}$ . The CNF was dispersed in deuterated water; the dispersion was subsequently sprayed on a cleaned, hydrophilic silicon substrate. Using an environmental cell, we investigated the uptake of deuterated water in the 200 nm CNF thin film. We present here initial GISANS results on the nanostructural changes during hydration and drying of the CNF thin films.

[1] Håkansson et al., Nat. Commun. 5, 4018 (2014); [2] Roth, J. Phys.: Condens. Matter 28, 403003 (2016)

## Nuclear Inelastic Scattering and Density Functional Theory Studies of Spin Crossover Compound ligand $[\text{Fe}(1,2,4\text{-triazole})_2(1,2,4\text{-triazolato})](\text{BF}_4)_2$

ID 187

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Since it has been synthesized [1], the  $\text{Fe}(\text{trzH})_2(\text{trz})$  complex is one of the most investigated spin crossover systems [2]. It contains the 1D polymeric chains and reveals the spin transition with the hysteresis of 40 K. In order to understand its vibrational properties we performed Nuclear Inelastic Scattering (NIS) experiments at the temperatures corresponding to its high-spin and low-spin states [3]. The DFT calculations for linear oligonuclear ( $n=7,9$ ) models of the compound yielded the normal vibrations and electronic energies for high-spin and low-spin isomers of three different models, differing in the distribution of anionic trz- ligands and  $\text{BF}_4^-$  anions. On the basis of the obtained energies the structure exhibiting the centrosymmetric  $\text{Fe}(\text{trzH})_4(\text{trz})_2$  coordination core for each but the terminal irons is proposed. The distribution of the  $\text{BF}_4^-$  anions in the structure is close to that obtained on the basis of X-ray powder diffraction studies by Grosjean et al [4]. The NIS spectra of the parent complex diluted in the matrix of the corresponding  $\text{Zn}(\text{II})$  complex show the change of the spectral pattern of the low-spin centres. That is shown, on the basis of the DFT calculations, to be the result of the change of the structure of the neighbours.

1. J. Kröber et al, Chem. Mater., 6, 1404 (1994).
2. A. Bousseksou et al. Chem. Soc. Rev., 40, 3313 (2011).
3. K. Jenni et al. Phys.Chem.Chem.Phys., 19, 18880 (2017).
4. A. Grosjean et al. Eur. J. Inorg. Chem., 796 (2013).



## Neutron optics for neutron beta decay studies with Proton Electron Radiation Channel (PERC)

ID 190

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The PERC experiment is currently under construction at the new beam port MEPHISTO at the FRM II. It aims to measure correlation parameters in neutron beta decay with an accuracy improved by one order of magnitude to a level of  $10^{-4}$ . We will present an overview of the demanding experimental constraints for this precision experiment with a focus on the beamline with its neutron optical components. In particular, this experiment requires a completely non-depolarizing supermirror coating made from Copper/Titanium. We present the current status of its development of as well as results for a solid-state neutron polarizer made from Iron/Silicon layers.

## Printing technology for photovoltaic applications

ID 191

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Organic solar cells (OPVs) are a very promising technology of photovoltaics and exhibit advantages in comparison to inorganic photovoltaics, like light weight, flexibility, low-cost processing and lower energy payback time. Due to this, they have been under intense investigation over the last decades. To make them attractive for industrial application and production, it is essential to ensure the competitiveness of the OPVs by increasing its reliability and efficiency as well as improving and simplifying its fabrication process. Commonly, organic solar cells in lab scale are prepared using the spin coating method. Other methods, suitable for industrial application, are spray coating or printing. Both methods have the advantage of a large-scale production, especially the inexpensive roll-to-roll printing exhibits one of the best prospects to process OPVs on large surfaces. In this study, investigations on the printing technology for organic materials applied on flexible substrates are carried out. With the printing technology the fabrication process of OPVs are intended to be optimized. Further, photoelectric characteristics as well as spectroscopic measurements are performed to receive deeper insight into the correlation of the different properties of printed organic solar cells. The film morphology is probed with scattering methods.



## Conformational dynamics of proteins studied by time resolved small angle X-ray scattering combined with THz irradiation

ID 193

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Proteins rely on conformational changes to perform complex biochemical tasks. This dynamic plasticity can be modeled by large scale vibrational modes that have theoretical resonance frequencies in the range of 0.3 to 6 THz and can therefore be triggered by THz radiation. Since these modes are associated with conformational changes of several Angstrom to nm, they can be probed by small angle X-ray scattering (SAXS). We aim to observe synchronized collective vibrational modes in time resolved SAXS experiments with external THz excitation. Apart from standard protein samples, we aim to investigate porcine microtubules and human dipeptidyl peptidase 3 (DPP3). These systems exhibit large scale conformational plasticity and characteristic SAXS profiles. The major challenge for this type of experiment is the sample environment. The harsh conditions under X-ray irradiation require constant material flow during the SAXS experiment. Simultaneous irradiation with THz radiations requires a thin liquid film ( $< 200\mu\text{m}$ ) to compensate for the high absorption coefficient of aqueous solutions in the THz regime. Furthermore, window materials for X-ray and THz transmission have to be chosen with great care to avoid excess absorption or scattering artefacts. We design flow cells that fulfill these requirements to enable combined SAXS-THz experiments.

## An XUV and soft X-ray split-and-delay unit for FLASH II

ID 196

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An XUV and soft X-ray split-and-delay unit is built that enables time-resolved experiments covering the whole spectral range of FLASH II from  $h\nu = 30$  eV up to 2500 eV. With wave front beam splitting and grazing incidence angles a maximum delay of  $-6 \text{ ps} < \Delta t < +18 \text{ ps}$  will be possible with a sub-fs resolution. Two different coatings are required to cover the complete spectral range. Therefore, a design that is based on the three dimensional beam path of the SDU at BL2 at FLASH has been developed which allows choosing the propagation via two sets of mirrors with these coatings. A Ni-coating will allow a total transmission on the order of  $T = 55 \%$  for photon energies between 30 eV and 600 eV at a grazing angle  $\theta = 1.8^\circ$  in the variable delay line. In the fixed delay line the grazing angle is set so  $\theta = 1.3^\circ$ . With a Pt-coating a transmission of  $T > 13 \%$  will be possible for photon energies up to 1500 eV. For a future upgrade of FLASH II the grazing angle can be changed to  $\theta = 1.3^\circ$  in order to cover a range up to  $h\nu \approx 2500$  eV.

## Upgrades of the neutron scattering instruments at MLZ for soft matter research

ID 200

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Many interesting scientific proposals for soft matter research are submitted to MLZ twice a year, and lead to ca. 30% of the scientific output. The soft matter group at MLZ practices also own in-house research that is tightly connected to TUM & LMU activities and strongly supported by Forschungszentrum Jülich with its activities. This concept with active research enables the soft matter group to drive the instrument development towards the future scientific needs with continuously raising complexity. The mostly soft matter related instruments are the small angle diffractometers KWS-1, -2 and -3, the spin echo spectrometer J-NSE, the backscattering spectrometer SPHERES, and the reflectometer MARIA. The instruments KWS-1 and -2 are equipped with large area detectors from GE/Reuter-Stokes that count several MHz of neutrons. Furthermore, polarization analysis allows to precisely subtract the unwanted incoherent scattering. The J-NSE instrument is furnished with superconducting coils that reduce the statistic noise to the lowest level worldwide. SPHERES suppresses unwanted background with a new chopper. MARIA with its velocity selector serves for highest intensities in the field. The sample environment for all instruments is continuously developed. For instance microfluidics will support the demand for smallest sample volumes that often limit conventional experiments. With this new instrument suite, the MLZ is prepared for the demanding soft matter experiments of the next decade.

## Analysis of H<sub>2</sub>O/D<sub>2</sub>O interaction with PNIPAM microgel thin films during swelling and exchange kinetics

ID 201

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Microgels out of thermoresponsive polymers show a strong change in volume by a variation of the temperature across the lower critical solution temperature (LCST), which in a thin film geometry translates into a change of the film thickness. This offers new functionality and is of high interest in many different fields of application such as drug delivery or tissue growth. In order to gain detailed insights in the kinetics during the volume change, the diffusion of water molecules through the interconnected polymer network during the uptake and release of water is analyzed. Thin PNIPAM microgel films crosslinked with N,N'-methylenebisacrylamide are investigated with time-of-flight neutron reflectometry (TOF-NR) and Fourier transform infrared (FTIR) spectroscopy. H<sub>2</sub>O and D<sub>2</sub>O are used to apply different humidity conditions at a constant temperature below the LCST of PNIPAM and assure proper contrast between the low molecular penetrants for both measurement techniques. The H<sub>2</sub>O and D<sub>2</sub>O swelling as well as the H<sub>2</sub>O – D<sub>2</sub>O and D<sub>2</sub>O – H<sub>2</sub>O exchange processes are studied. Results from the TOF-NR analysis and the FTIR spectroscopy measurements are modelled with a model which accounts for the temporal evolution of the humidity and the diffusion speed of the low molecular penetrants through the polymer network.

## Nanostructured Mixed-Dimensional Lead Bromide Perovskite Films with Preferential Crystal Orientation Investigated with Advanced Scattering Techniques

ID 204

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Organometal halide perovskite materials have attracted tremendous interests in optoelectronic applications due to their intrinsic dielectric properties. To further boost these materials in to large scale application, a deep understanding of morphology-property correlation in perovskite films is essential. Particularly, it is challenging to thoroughly determine the complex film morphology. In the present work, lead bromide perovskite films composing of a mixture of threedimensional (3D) and two-dimensional (2D) perovskite crystals are prepared using a succinctly solution-processed route. The film morphologies are quantitatively characterized by grazing incidence small angle/wide angle x-ray and neutron scattering (GISAXS/GIWAXS and GISANS). The findings reveal that the resulting optoelectronic property closely interrelates with the tailored film nanostructures. A compact film morphology associated with an anisotropic preferential crystal orientation, as well as efficient energy transfer between 2D perovskite platelets and 3D perovskite bulk crystals secure a comparable low amplified spontaneous emission (ASE) threshold of 17.8  $\mu\text{J}/\text{cm}^2$ . This result renders the potential of realizing these materials for optical gain media in the future.

## Fine-tuning protein phase transitions: a TR-USAXS and VSANS study

ID 207

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Multivalent cations induce complex phase transitions in negatively charged globular proteins, e.g. reentrant condensation and liquid-liquid phase separation [1] with a lower critical solution temperature (LCST-LLPS) [2] featuring a strong dependence on the type of multivalent cations used [4]. Improved opportunities at large-scale facilities allow for detailed studies of the kinetics of LCST-LLPS in the above systems with complementary VSANS and TR-USAXS [3,4] in the low- $q$  region down to  $4 \times 10^{-5} \text{ \AA}^{-1}$  [5,6], resolving even big structures. Here, we present a systematic TR-USAXS and VSANS study of the kinetics of phase separation in protein-cation systems with varying interprotein interaction strengths. At low temperatures and with weak interactions, the temperature-dependent characteristic length scale of phase separation  $\xi$  grows as a function of time as  $\xi = (2\pi)/q \sim t^{1/3}$ . Increasing the interprotein interaction strength leads to strong deviations from this classical growth behaviour. We therefore show that an appropriate choice of the multivalent cations used can finetune phase transitions in protein systems.

- [1] Zhang et al., Pure Appl. Chem. 2014 (86), 181-202
- [2] Matsarskaia et al., JPCB 2016 (120), 7731-6
- [3] Da Vela et al., Soft Matter 2016 (12), 9334
- [4] Matsarskaia et al., in preparation, 2018
- [5] Pipich & Fu, JLSRF (1), 2015
- [6] Van Vaerenbergh et al., AIP Conference Proceedings 2016 (1741), 030034

## Time-resolved Structural Analysis of Solvent Vapor Annealing Processes of the Photoactive Material DRCN5T:PC71BM Using GIWAXS

ID 208

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The control of the micro- and the nanostructure in photoactive layers of organic photovoltaics is one key component for the performance of such devices. In this regard, we will present timeresolved grazing incidence wide angle scattering (GIWAXS) measurements of the photoactive material DRCN5T:PC71BM during solvent vapor annealing (SVA). In the course of this annealing process a phase separation of the donor (DRCN5T) and the acceptor (PC71BM) occurs and therefore the crystallinity of the donor increases greatly, as can be seen by the rise of several sharp Bragg peaks. This structural changes can lead to an increase in power conversion efficiency (PCE) from 3.57% to 6.64% [1]. We will discuss the influence of the solvent choice in respect to the donor or acceptor-selectivity of the solvent. In addition we will present a crystallographic structure determination, i.e. space group and lattice parameters, and compare it with complementary techniques (e.g. energy-filtered electron diffraction, ex-situ X-ray pole figures). The in-situ GIWAXS measurements were performed at the synchrotron beamline P08 at PETRA III (DESY) using our self-designed, specialized SVA- and doctor-blading cell, which will also be described.

[1]: J. Min et al., 2017, DOI: 10.1039/c7ta04769j



## Implementation of the Cryo-EXAFS environment at the XANES end station of the KMC-2 beamline

ID 210

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The KMC-2 [1] beamline at BESSY II [2], operates a graded SiGe monochromator (energy resolution  $E/\Delta E = 4000$ ; energy range: 4 – 15 keV). The newly build Cryo-EXAFS environment at KMC-2 “XANES” end station has been developed for in situ gas absorption combined with X-ray powder diffraction experiments [3] and has been modified for use on the XANES end station. The Cryo-EXAFS is based on a Gifford-McMahon (GM) closed cycle cooler system. Exchange gas atmosphere of up to 150 kPa or vacuum can be utilized as sample environment. Continuous gas flow or controlled volumetric gas adsorption infrastructures are available. With an integrated heater stage the temperature range 15 - 450 K is accessible. Due to the double-dome construction for isolation vacuum and heat-exchange gas (Helium) temperature gradients in powder samples are very small. With multiple Kapton windows and a variable sample holder system, experiments can be conducted in both fluorescence and transmission geometry. The Cryo-EXAFS cryostat is mounted inside a three-axis translation motor stage for fine tuning of the sample position. Temperature readings and regulation, realized through a Lakeshore LS-336 controller is fully integrated into the instrument control software spec (CSS), allowing for fully automated temperature profile collection.

[1] Journal of large-scale research facilities 2, A49 (2016) doi: 10.17815/jlsrf-2-65

[2] helmholtz-berlin.de

[3] S. Krause et al., Nature, 532 (2016) 348



## Diffraction Based Determination of Single Crystalline Elastic Constants on Polycrystalline Alloys

ID 211

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The tensor components of the stiffness tensor, also known as elastic constants, are directly related to material parameters such as the shear-, Young's- and Bulk modulus. But also non-mechanical properties such as the Debye temperature can be calculated using the tensor components. Elastic constants are essential for many applications, e. g. the stress analysis by diffraction, where from measured strains residual stresses are determined with use of the elastic constants. Different diffraction techniques allow the investigation of poly-crystalline and multi-phase materials due to its outstanding possibility to visualize the different strains of all phases averaged over all orientations. Combining this with the knowledge of the occurring stresses enables a type of reversal of the classical stress analysis calculation with elastic constants as result. The validation of our approach was done on different types of Iron S 235 JR (BCC), V2A (FCC) and Duplex Steel (BCC and FCC) revealing a good agreement between our results and the literature data. Further Investigations were performed on different technically applied Ti-alloys including  $\alpha$ -,  $\beta$ - and  $\alpha+\beta$ -Ti. Despite their technical relevance only a few comparable literature data exists. The correctness of the results was then ensured by calculating macroscopic values and comparing them to either literature data and/or own experiments.

## Phase transformations during battery operation in vanadium phosphate cathode materials probed by operando synchrotron X-ray diffraction

ID 212

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Phosphate based cathode materials has seen great success following the demonstration of  $\text{LiFePO}_4$  as a functioning Li-ion battery cathode material, especially due to its very low cost and high safety compared with the previously used oxide based materials. However, despite the inductive effect of the phosphate group,  $\text{LiFePO}_4$  has a relatively low discharge potential compared with materials based on other transition metals, such as vanadium, cobalt and nickel. The capacity is also low due to the limited number of oxidation states for iron, yielding only a single Li-ion per formula unit. An interesting alternative to  $\text{LiFePO}_4$  is  $\text{Li}_3\text{V}_2(\text{PO}_4)_3$  as it has the highest gravimetric capacity among the known phosphates ( $197 \text{ mAh g}^{-1}$ ). The material displays a complex series of phase transformations during charge and discharge, and interestingly, these transformations are very dependent on the number of Li-ions extracted during charging. In recent years, new methods have been developed to investigate the dynamic structural behavior of battery materials during operation using synchrotron X-ray diffraction. The methods are expected to give a more realistic view of the behavior of batteries in operation. In this study, we investigate the dynamic structural behavior of  $\text{Li}_3\text{V}_2(\text{PO}_4)_3$  as a function of extracted Li-ions using in-situ synchrotron diffraction. The detailed structural information of the phase transformations in this material clearly demonstrates the strength of in-situ diffraction methods.

## Practical Aspects of Crystallographic Fragment Screening

ID 214

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Crystallographic fragment screening is an established method to initiate structure-based compound development and has become feasible for a wide scientific community. Our efforts within Frag2Xtal and Frag4Lead projects enable users to carry out these experiments in practicable time at the Helmholtz-Zentrum Berlin. Efficient soaking and handling of crystals is supported via our Frag2Xtal Screen (made available in collaboration with Jena Bioscience) and our Easy Access Frame tool (patent pending). Furthermore, robot-assisted beamlines with high sample changing rates, automated data processing and refinement of the protein-ligand structure models decrease manual labor. With our 12-compound pre-screen, users can get accustomed to the soaking optimization and crystal fishing procedure and are guided towards successful campaigns. Additionally, our collaboration partners from the drug design group in Marburg composed a 1270 fragment library based on advanced clustering to balance 3D-diversity versus pharmacophore representation. Currently, we establish handling of the library and providing ready-to-soak plates for users by utilizing an acoustic dispenser. Together, we are also advancing the follow-up methods for successfully identified fragment hits by employing our new Frag4Lead web service as well as molecular docking to choose purchasable, diversified compounds for downstream lead optimization. We present results of example campaigns and subsequent compound diversification.

## In-situ GISAXS during sputter deposition of metal nanolayers on functional polymer thin films for lithium-ion batteries

ID 217

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Understanding the interface between metals, commonly used as current collectors, and ion-conducting polymers used in polymer lithium-ion batteries (LIBs) is crucial to develop highly reproducible, low-cost and reliable devices. To address these issues, sputter deposition is the technique of choice to fabricate scalable, reproducible and controllable nanometer and sub-nanometer metal layers on polymer thin films. The sputter deposition process, being well understood and controlled, offers advantages over chemical methods to tailor metal thin-film morphologies on the nanoscale and offers a superior adhesion of the deposited material.[1] We use in-situ grazing incidence small angle X-ray scattering (GISAXS) to investigate the formation, growth and, self-assembled structuring on polymer thin films used in LIBs.[2] The growth of noble metal layers on different polymer films is investigated.

[1] Schwartzkopf et al., ACS Appl. Mater. Interfaces 9, 5629 (2017).

[2] Schwartzkopf et al., ACS Appl. Mater. Interfaces 7, 13547 (2015).



## Advanced gas atmosphere options at the BESSY II beamline KMC-2

ID 221

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At the Berlin synchrotron BESSY II, beamline KMC-2 provides two permanent stations with intensity stabilized radiation in the energy range of 4-14 keV: "XANES" for EXAFS and XRF, and "DIFFRACTION", a multi-purpose six-circle goniometer. A wide range of sample environments is available for both stations [1]. Of particular interest is a suit of systems for controlled gas atmosphere: Low pressure (< 130 kPa) volumetric gas adsorption is possible with the multi-purpose gas rig CGA-GH11 or, for in-situ sorption isotherm measurements, with a fully automated BELSORP gas dosing system. Alternatively, CGA-GH7 is an automated continuous gas flow and pressure control system for pressures up to 30 MPa. CCR-XRD, a GM closed-cycle cryocooler equipped with a high temperature stage covers temperatures 15-450 K and controlled gas atmospheres from vacuum up to 150 kPa. Both transmission or reflection at the sample are possible. For small samples or precarious gases, in continuous flow or static atmosphere up to 10 MPa, several glass capillary setups are available. Dedicated temperature environments cover a range 20-700 K, preventing moisture condensation and temperature inhomogeneities, while allowing sample rotation for enhanced particle statistic. A new system for humidity control is currently under development in cooperation with TU Dresden. All systems are available to users of KMC-2 @ BESSY II.

[1] [www.helmholtz-berlin.de/user/experimental-infrastructures/sample-environment/se-at-bessy/](http://www.helmholtz-berlin.de/user/experimental-infrastructures/sample-environment/se-at-bessy/)

## Gold Cluster Growth on Polymer Thin Films during Sputter Deposition

ID 222

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Fabricating nanocluster films with tailored morphology, their characterization and manipulation at the nanoscale are essential prerequisites for more efficient solar cells, biosensors, reflective or antireflective coatings, and heterogeneous catalyst. In order to tune the size-dependent optoelectronic properties, it is mandatory to monitor how the growth kinetics affect the metal film morphology and how it correlates to the optical properties during sputter deposition. We employed a combination of in situ time-resolved Grazing Incidence Small Angle X-ray Scattering (GISAXS) with in situ UV/Vis Specular Reflectance Spectroscopy (SRS) during sputter deposition of gold on thin polystyrene films [1]. We monitored the evolution of the metallic layer morphology according to changes in the key scattering features by geometrical modeling [2] and correlate the nanostructural development to optical properties. This enables us to identify the different growth regimes including their specific thresholds with subnanometer resolution, which permits a better understanding of the growth kinetics of gold clusters and their self-organization into complex nanostructures on polymer substrates. During sputter deposition, a change in optical reflectivity of the pristine grey-blue polymer (polystyrene, PS) film occurred ranging from dark blue color due to the presence of isolated nanoclusters at the interface to bright red color from larger Au aggregates [3]. Furthermore, a surface diffusion coefficient according to the kinetic freezing model and interfacial energy of Au on PS at room temperature were calculated based on a real-time experiment. A recent study reports on the role of sputter deposition rate in tailoring metal layer morphologies on polymer thin films [4]. The deposition rate affects primarily the nucleation process and the adsorption-mediated growth, whereas rather small effects on diffusion-mediated growth processes are observed. Only at higher rates, initial particle densities are higher due to an increasing influence of random nucleation and an earlier onset of thin film percolation occurs.

[1] Schwartzkopf M and Roth SV; *Nanomaterials*, 6, 239 (2016).

[2] Schwartzkopf M et al.; *Nanoscale*, 5, 5053-5062 (2013).

[3] Schwartzkopf M et al.; *ACS Appl. Mater. Interfaces*, 7, 13547-13556 (2015).

[4] Schwartzkopf M et al.; *ACS Appl. Mater. Interfaces*, 9, 5629-5637 (2017).

## Emergence of cooperativity in the coverage-dependent spin transition of an Fe(II) complex on a graphite surface

ID 223

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The spin-state manipulation of spin crossover molecules (SCMs) adsorbed on a surface between a paramagnetic high-spin state and a diamagnetic low-spin state has attracted significant interest due to its potential for application in spintronic devices. The absence or presence of cooperativity in the spin transition and their characteristics is one of the most important issues for research, since it is responsible for the bistability in the spin states of SCMs. We present temperature- and coverage-dependent x-ray absorption spectroscopy measurements obtained at the VEKMAG and the high-field diffractometer end stations at BESSY II of the complex  $[\text{Fe}(\text{H}_2\text{B}(\text{pz})_2)_2(\text{bipy})]$  deposited on a highly oriented pyrolytic graphite surface, investigating the ultimate scale limit at which cooperative spin switching becomes effective. This system exhibits a complete thermal- and light-induced spin transition. On increasing the coverage, the width of the temperature-induced spin transition curve narrows significantly, evidencing the buildup of cooperative effects. While the molecules at submonolayer coverage exhibit an apparent anticooperative behavior, the multilayers starting from a double-layer exhibit a distinctly cooperative spin switching. These observations will serve as useful yardsticks in designing SCM-based devices.



## Oxygen Vacancies in High-Tc Superconductor Studied with a Scanning Positron Beam

ID 224

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In solid state physics and materials science the positron is applied as a highly mobile nano-probe for the detection of vacancy-like defects and their chemical surrounding using (coincident) Doppler broadening spectroscopy ((C)DBS) of the positron-electron annihilation line. A scanning positron beam allows depth dependant defect spectroscopy and the imaging of defect distributions. The neutron induced positron source NEPOMUC at FRM II provides the world's highest intensity of 109 moderated positrons per second. Within this contribution we present spatial resolved (C)DBS experiments on thin film samples of the high-Tc superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (YBCO). First, the positron beam was applied to image the oxygen deficiency. By combining transport measurements, X-ray diffraction and CDBS we succeeded in correlating the relevant parameters  $T_c$ , expansion of the c-axis, oxygen deficiency  $\delta$ , and the S-parameter obtained by DBS [1]. Afterwards, the positron implantation energy was varied in order to reveal the depth dependent transition temperature in tempered thin film YBCO samples [2]. Financial support from the BMBF within the projects 05K13WO1 and 05K16WO7 is gratefully acknowledged.

## Influence of shear forces on the structure and flow behaviour of casein micelles

ID 228

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Casein micelles (CM) are complex, highly hydrated colloidal particles of spherical shape in milk with many structural similarities to microgels. During processing, CM are subjected to a wide range of shear stress whose effects on molecular structure are unknown. Viscosity curves of CM suspensions were measured over a broad temperature and concentration range. The measured relative viscosity increased with increasing concentration and decreasing temperature. CM suspensions with  $w \leq 0.11$  behaved like Newtonian fluids. Above this concentration, shear thinning was observed with Newtonian behaviour at low and high shear range. The experimental data were fitted in good approximation with a viscosity model for microgels. Additionally, the flow behaviour of CM is determined over a wide range of milieu and process conditions (e.g. ionic strengths and pH values), fitted with the above mentioned viscosity model for microgels and explained by experimentally proven molecular structural changes. For this purpose, Rheo-SANS experiments are used for the first time in the area of CM research to gain structural information depending on shear stress and to validate the limits of the viscosity model for microgels. Rheo-SANS experiments will be carried out in dependence of selected concentrations, temperatures, ionic strengths and pH/pD values.

## Sub-micrometer angle-, spin-, and polarization-dependent photoelectron spectroscopy in the tender X-ray regime

ID 230

**KALLAENE, Matthias**

*BENTMANN, Hendrik; QUER, Arndt; Mr ROHLF, Sebastian; DIEKMANN, Florian; RIEDEL, Tim; ROSSNAGEL, Kai (Kiel University)*

ASPHERE III is an angle-, spin-, and position-resolving photoelectron spectrometer that fully exploits the unique capabilities of the variable polarization tender X-ray beamline P04 of PETRA III. Combining a Scienta DA30 photoelectron analyzer with installed 3D-Mott detector and the brilliant, widely tunable (250–3000 eV), and highly monochromatic photon beam of the beamline or, complementary, with the monochromatized beam of a VUV-He- and Xe-plasma source, the experimental setup enables a direct comparison between bulk and surface electronic structures of solids. Employing the DA30 deflector driven angle mapping mode and the UHV goniometer that rotates the analyzer around the sample, complete three-dimensional band structures and Fermi surfaces without sample rotation can be determined efficiently. After the permanent integration of ASPHERE III into the beamline sector in 2019 the spatial resolution is aimed to be in the sub micrometer range and will be available for all ASPHERE III related spectroscopic methods. To connect the measured spin-, momentum- and position-resolved electronic structure directly with the geometric structure, additional XPD investigations from the same spot on the sample and STM and LEED investigations from the same sample surface can be performed. All spectroscopic techniques are available during sample temperature changes controlled with a He-flow cryostat as well as during metal deposition on sample surfaces.



## Professionalizing scientific software development

ID 231

*WUTTKE, Joachim (JCNS at MLZ)*

Most software in our field has been developed by single researchers. This is no longer sustainable. We need software that is continuously improved and extended, runs on different platforms, is fully documented and covered by tests, and has a friendly user interface. This can only be achieved if software development is institutionalized. All major neutron facilities have recognized this need, and build up scientific computing groups. In this talk, I will review the first seven years of the Scientific Computing Group of MLZ. I will emphasize the importance of open-source principles and international cooperation, and present the division of labor agreed upon in SINE2020. I will then give a brief overview of our current projects in the field of reflectometry and GISAS, single crystal diffraction, and materials diffraction.

## Swift heavy ion research of condensed matter at extreme conditions

ID 233

**FALLER, Debora (Universität Heidelberg)**

*BURCHARD, Michael (Institute of Earth Sciences, Heidelberg University); GLASMACHER, Ulrich A. (Institute of Earth Sciences, Heidelberg University); TRAUTMANN, Christina (GSI Helmholtzzentrum für Schwerionenforschung GmbH)*

Research at extreme P-T conditions forces the available technologies and material properties to their limits. The Paris-Edinburgh press (PE press) yields a suitable technology for generating stable static pressures (10's of GPa) on large sample volume (< 3mm<sup>3</sup>). In the past, two PE presses have been modified to such a degree that accelerated heavy ions can reach condensed matter held at high pressure. The specially developed anvils manufactured of tungsten carbide enclose new one-side spherical diamonds (diamond diameter = 2.7 mm). These diamond windows open the pathway for optical and spectroscopic measurements during ion irradiation. With this novel originated press setup, it is possible to retrieve pressure and temperature data by measuring the shift of Raman bands or fluorescence lines of standard material. The poster will provide insight into the latest technological developments. One of the PE presses has been modified to perform swift heavy ion irradiation of condensed matter at high pressure and medium high temperatures (<900 K). The second PE press will be customized to operate at medium low temperatures (200 - 300 K). Currently, the PE cell with a heating set up is undergoing feasibility tests. In October, also investigations with simultaneously ion irradiation are planned at the Cave A at GSI, Darmstadt. The aim of the research is to conduct experimental studies in such a way that radioactive decay and its effect to Earth mantle rocks at HT/HP-conditions can be simulated. In addition, the low temperature research aims to simulate the interaction of cosmic radiation on ice on planetary surfaces in space.

## Super-SIMS at HZDR - first steps

ID 237

**RUGEL, Georg (Helmholtz-Zentrum Dresden-Rossendorf)**

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The integration of an ion source with very high spatial resolution with a tandem accelerator is a long-standing concept for improving analytical selectivity and sensitivity by orders of magnitude [1-3]. Translating this design concept to reality has its challenges [e.g. 4-6]. Supporting a strong focus on natural, metallic and mineral resources the, Helmholtz Institute Freiberg for Resource Technology installed such a system at the Ion Beam Centre at HZDR. This so-called Super-SIMS will be at the core of a comprehensive pallet of micro-analytical methods devoted to the characterization of minerals and ores. Secondary ion beam from a CAMECA IMS 7f-auto are injected into the pre-existing 6MV Dresden Accelerator Mass Spectrometry facility [7,8], which quantitatively eliminates isobaric molecular species from the ion beam. Our SIMS component can function as either a stand-alone device or can be used to inject the negatively charged secondary ions at energies of up to 40 keV (to match the acceptance conditions) into the accelerator. A dedicated ion optical unit has been constructed and installed to match the SIMS ion beam to the maximum acceptance of the accelerator. We will present measurements of the performance parameters of the instrument as well as first results of halogen (F, Cl, Br, and I) determinations in galena, sphalerite and pyrrhotite.

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## Pore-scale Imaging of Oil Flow Dynamics In a Mixed-wet Carbonate Reservoir Rock at Subsurface Conditions Using Synchrotron Fast Tomography

ID 240

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**SINGH, Kamaljit** (Department of Earth Science and Engineering, Imperial College London); **Dr LIN, Qingyang** (Department of Earth Science and Engineering, Imperial College London,); **SCANZIANI, Alessio** (Department of Earth Science and Engineering, Imperial College London); **BLUNT, Martin** (Department of Earth Science and Engineering, Imperial College London); **BIJELJIC, Branko** (Department of Earth Science and Engineering, Imperial College London)

Imaging and characterization of multiphase flow through porous media at the pore-scale is essential to understand the fluid dynamics that control for instance oil recovery and efficiency of carbon dioxide storage in the subsurface formations. Non-destructive dynamic pore-scale imaging using X-rays generated by a synchrotron light source along with an advanced flow apparatus that is almost transparent to X-rays and can withstand high pressures and temperatures have allowed the imaging of fluid dynamics in porous media at subsurface conditions. In this work, an aged carbonate reservoir rock (mixed-wet) extracted from a very large producing oil field was fully saturated with prepared reservoir formation brine. Then, oil (crude oil mixed with 25 weight percent of diiodomethane used as an X-ray dopant) was injected at constant flow rate (2  $\mu\text{L}/\text{min}$ ) at subsurface conditions (10 MPa and 60°C). The dynamics of oil invasion through the mixed-wet reservoir rock were captured by fast X-ray three-dimensional imaging while measuring the pressure drop across the rock sample using a very sensitive pressure transducer. Oil percolated through the centre of larger pores with oil-wet surfaces. Whereas, brine was retained in pore corners and interstices that were water-wet. When oil reached the hydrophilic porous plate at the base of the rock sample, the pressure drop started to build up resulting in oil invasion into smaller pores that have higher capillary entry pressure.

## The role of entropy in organic-inorganic perovskites and related coordination networks

ID 242

**KIESLICH, Gregor (Technical University of Munich)**

Over the past decades, crystalline organic-inorganic network materials such as hybrid inorganicorganic perovskites (HOIPs) and metal-organic frameworks (MOFs) have changed our perception of the solid state. Such materials bring many opportunities in application-oriented research areas such as optoelectronics, photovoltaics, catalysis and gas sensing among others. At the same time, the large amount of relatively weak molecular interactions such as hydrogen bonds and dispersion interactions make materials design a challenging task. This becomes even more challenging when interested in understanding phase transitions in such materials as a function of temperature and pressure that are related to structural flexibility as observed in flexible MOFs or for ferroelectric phase transitions in HOIPs. Relatively low densities in inorganic-organic network materials facilitate the role of lattice entropy – a parameter that is barely discussed in traditional rigid-body guidelines of organic-inorganic network materials.[1,2] In my presentation I address the role of entropic contributions in organic-inorganic coordination networks. I draw on topical examples where state-of-the-art synchrotron methods such as variable temperature high-resolution powder X-ray diffraction and Neutron Inelastic Scattering have been used to insight the thermodynamics of organic-inorganic materials. For instance, the phase evolution as function of temperature in  $[\text{CH}_3\text{NH}_3]\text{PbBr}_3$  is discussed, identifying configurational entropy, lattice entropy and hydrogen bonding interactions as the critical parameters. Similar conclusions can be drawn for flexible MOFs such as ZIF-4 and  $[\text{M}_2(\text{bdc})_2(\text{dabco})]$  with  $\text{M} = \text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  in which the balance of dispersion interactions and lattice entropy determines the underlying thermodynamics of these exciting materials.

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## The footprint from distinctly rough gratings in the scattering pattern

ID 243

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*PFLÜGER, Mika* (*Physikalisch-Technische Bundesanstalt*); *SOLTWISCH, Victor* (*Physikalisch-Technische Bundesanstalt*); *SCHOLZE, Frank* (*Physikalisch-Technische Bundesanstalt*)

For the control of the lithographic manufacturing process, a rapid in-line metrology is needed. New metrology solutions may accomplish the characterization of more complex 3D structures, where the effect of the roughness becomes even more acute. Grazing incidence small angle X-ray scattering (GISAXS) is a reliable technique with high surface-sensitiveness which allows short acquisition times. However, the footprints obtained are much larger than the structured area. We counteract this disadvantage by using larger incidence wavelengths in the EUV-photon energy range, which allows larger incidence angle without compromising the surface sensitiveness, for the investigation of rough lamellar gratings. We investigate the contributions due to the roughness to the scattered pattern. Therefore a set of gratings was designed with a controlled roughness. Two different types of roughness are distinguished: line edge roughness (LER), where the center position changes along the line and line width roughness (LWR), where the width of the line changes and different types of roughness distribution: periodic, chirped or stochastic. Each type of roughness leads to a different scattering pattern, which opens new perspectives for the unequivocal characterization of such structures by EUV-scatterometry.

## On the electronic and geometric structure of a single atom Cu/UiO-66 CO oxidation catalyst during reaction - An operando XAFS study

ID 244

**ABDEL-MAGEED, Ali (Ulm University)**

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The single atom Cu metallated UiO-66 metal organic framework (MOF) catalyst (Cu/UiO-66) had turned out to be highly active for CO oxidation at reaction temperatures between 120 and 250°C. Combining time resolved operando XAS measurements at the Cu K-edge with operando FTIR spectroscopy, atomic resolution electron microscopy (HAADF-STEM) and DFT computations we investigated the structure of this catalyst during reaction. The measurements indicated that it reaches its highest activity after an initial activation period of 800 min, then it remains stable at extended reaction time and also under dynamic startup-shutdown operations. While XANES measurements showed that Cu exists in a composition of 30 % Cu<sup>1+</sup> and 70% Cu<sup>2+</sup> under steady-state conditions, EXAFS measurements indicated that Cu is predominantly bound to O, with an average coordination of  $2.0 \pm 0.5$  at Cu-O bond distance of  $1.90 \pm 0.02$  Å. In agreement with these findings, electron microscopy (HAADF-STEM) indicated that Cu exists in a highly dispersed atomic state. Consequences of these findings and of additional DFT computations for the understanding of the Cu@UiO-66 structure under reaction conditions and the mechanistic understanding of these catalysts will be discussed.

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2. B. Rungtaweevoranit et al., Nano. Lett. 16 (2016) 764.

## Structural Characterization of Organic Nanoparticles Prepared by Antisolvent Precipitation

ID 249

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The production of organic nanoparticles (NPs) via antisolvent precipitation (AP) is a less-invasive and low-cost alternative to top-down methods like high-pressure homogenization [1]. Thus, AP is receiving growing attention, e.g. for the preparation of nanodispersions of pharmaceutical ingredients [2]. For instance, the antioxidant coenzyme Q10 (Q10) has several health protective effects and Q10 NPs showed improved bioavailability compared to free Q10 [3, 4]. Here we report the structural characterization of Q10 NPs produced by AP. The NPs are analyzed by photon correlation spectroscopy (PCS), cryogenic transmission electron microscopy (CryoTEM), small-angle X-ray and neutron scattering (SAXS; SANS, KWS-1 at MLZ). In particular, the combination of SAXS and SANS studies allows the investigation of the molecular structure of the NPs and the interface between the NPs and the dispersion medium [5]. Our experiments revealed, that Q10 NPs prepared by AP are stable with and without added stabilizer, have a particular small size (down to ~ 20 nm in diameter), and exhibit a specific stabilizing layer, suggesting a self-stabilizing process of the Q10 molecules.

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## On the origin of Au charging during green methanol synthesis on Au/ZnO An in situ / operando spectroscopy study

ID 250

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Some Au catalysts, in particular Au/ZnO, have turned out to be highly active and selective for methanol synthesis from CO<sub>2</sub> and H<sub>2</sub>, a key reaction for chemical energy storage. Further improvement of their performance entails a molecular scale understanding of their performance. Here we report on reaction induced changes of structural and electronic properties of Au/ZnO, applying a combination of kinetic and time resolved in situ / operando spectroscopies including high-pressure operando FTIR spectroscopy, near ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) and X-ray absorption spectroscopy (XAS) at the O K-edge, in addition to temporal analysis of products (TAP) measurements. Operando DRIFTS measurements revealed that CO formed as side product during methanol synthesis adsorbs primarily on metallic or negatively charged gold sites. NAP-XPS and XAS measurements point to a close correlation between i) the formation of O-vacancies during reaction, ii) the methanol formation rate, and iii) the COad coverage on charged sites. TAP titration of Au/ZnO indicated that CO<sub>2</sub> can reversibly refill the O-vacancies created during reaction by CO or H<sub>2</sub>. In total, these results demonstrate a close correlation between the formation/replenishment of O-vacancies, charging of Au nanoparticles and the methanol formation activity.

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## The high resolution diffraction beamline P08

ID 256

**BERTRAM, Florian**

*KIRCHHOF, Rene (DESY Photon Science); BUSSONE, Genziana (DESY Photon Science); VOLKOV, Sergey (DESY Photon Science); SHEN, Chen (DESY Photon Science)*

The high resolution diffraction beamline P08 at Petra III is dedicated to high resolution X-ray diffraction and scattering methods with an emphasis on studies of surfaces, interfaces and thin films. The photon energy can be set between 5.4 and 29.4 keV with a focal spot between  $(20 \times 2) \mu\text{m}^2$  and  $(1 \times 0.5) \text{mm}^2$ . A high precision 6-circle diffractometer for solid samples and a diffractometer for scattering from liquid surfaces and interfaces are available at the beamline.

## Morphology of Amphiphilic Molecular Brushes in Dilute Aqueous Solutions

ID 264

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ZHAO, Junpeng (National Hellenic Research Foundation, Theoretical and Physical Chemistry Institute, Athens, Greece); PISPAS, Stergios (National Hellenic Research Foundation, Theoretical and Physical Chemistry Institute, Athens, Greece); PAPADAKIS, Christine M. (TU München, Physik-Department, Garching, Germany)

Molecular brushes are densely grafted polymers composed of a polymeric backbone and side arms. Their complex architecture can easily be adjusted by changing the side arm architecture. A series of amphiphilic molecular brushes with diblock or random copolymer side arms is investigated, where the side arms contain both poly(propylene oxide) (PPO) and poly(ethylene oxide) (PEO) segments, both having lower critical solution behavior in aqueous solution, leading to a conformational transformation of the side arms, and thus the molecular brushes. In the present work, the temperature-dependent conformation of the molecular brushes with PPO-PEO and PPO-*b*-PEO side arms in dilute aqueous solution is investigated by small-angle neutron scattering along with dynamic light scattering. As temperature is increased, the random brush shows a strong increase in size at a temperature of 41 degrees C, where they aggregate; for the block copolymer brush, the size increases gradually before the aggregation sets in at 68 degrees C. This model system provides insight into the effects caused by the t

## Ultrafast Demagnetization by Extreme Ultraviolet Light

ID 265

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Free-electron lasers (FELs) enable the study of dynamics in matter on combined femtosecond time and nanometer length scales [1]. One of the most intriguing topics within contemporary research on magnetism, ultrafast near-infrared (IR) laser-induced demagnetization [2,3], has greatly benefited from the advent of FELs [4] as it was shown that optically generated superdiffusive spin currents [5] contribute to that phenomenon [6]. Following a previous campaign [7], here we report on the observation of a breakdown of the magnetic scattering cross section of Co/Pt multilayers for extreme ultraviolet (XUV) fluences  $>1$  mJ/cm<sup>2</sup> defining the threshold fluence for FEL experiments where the FEL is meant to be a non-invasive probe. By employing a FEL double-pulse scheme, XUV-induced demagnetization is identified to be the major mechanism behind the breakdown. Besides revealing the existence of ultrafast demagnetization in the XUV regime for the first time, our results demonstrate that it proceeds much faster than the demagnetization when using IR radiation.

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- [7] L. Müller et al., Phys. Rev. Lett. 110, 234801 (2013).



## Exploration of dynamic fluid regimes during steady-state multiphase flow in a sandstone with using synchrotron imaging

ID 267

**GAO, Ying (Imperial College London)**

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The Diamond Lightsource Pink Beam was used to image dynamic fluid flow in Bentheimer sandstone at steady state, at a resolution of  $5.2 \mu\text{m}$  during the co-injection of oil and water together. We present a novel method that uses fast synchrotron tomography to examine flow mechanisms underground by observing oil and water distribution through time. Bentheimer is a homogeneous sandstone with a pore size from  $8$  to  $11 \mu\text{m}$ , whose connected porosity resides in the macro-porosity with very little micro-porosity. A non-wetting phase, decane, and a wetting phase,  $15 \text{ wt\% KI}$  brine, were co-injected at equal flow rates into a micro-core  $5 \text{ mm}$  across. Both fluids were injected simultaneously into the core and were collected at the outlet with a back pressure regulator. Tomographic scans were taken successively with a  $6.7 \text{ mm}$  by  $5.6 \text{ mm}$  field of view. Total acquisition time was  $\sim 1 \text{ min}$  per scan. The total flow rate increased from  $0.02 \text{ mL/min}$  ( $\text{Ca} \sim 2.5 \times 10^{-7}$ ) to  $2 \text{ mL/min}$  ( $\text{Ca} \sim 2.5 \times 10^{-5}$ ) step by step. We found that when  $\text{Ca}$  is lower than  $10^{-6}$ , oil and water reside their own pore space even when there is a sudden increase in flow rate. However, when  $\text{Ca}$  is higher than  $10^{-6}$ , an increase rate will alter the fluid distribution. The higher the flow rate, the greater the fluid rearrangement. Eventually a steady-state is reached, but even here some pore-space configurations continue to fluctuate.



## Magnetic skyrmions study in Fe(0.34nm)/Gd(0.45nm, 0.50nm, 0.55nm)]×80 multilayers

ID 269

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*BOENI, Peter (Technical University of Munich); ALBRECHT, Manfred (University of Augsburg); KREUZPAINTNER, Wolfgang (Technical university of Munich); MATTHIAS, Opel (Walther-Meißner-Institut); BENK, Georg (Technical university of Munich)*

Recently, the attention has shifted to ultrathin ferromagnetic/heavy metal films deposited by sputtering, allowing to explore how the balance of ferromagnetic exchange, anisotropy and dipolar energy results in cylindrical domains. Therefore, we have started to investigate Fe(0.34nm)/Gd(0.45nm, 0.50nm, 0.55nm)]×80 multilayers, which can reveal skyrmion lattices at room temperature. The magnetic phase diagram for these thin films displays that a broad skyrmion phase takes place around room temperature. In this project, we will explore the formation of dipole-stabilized skyrmion lattices in amorphous Gd/Fe based multilayers, with the focus to develop an understanding of the mechanisms that are responsible for the expected stabilization of chiral textures. For this purpose, the magnetic parameters will be controlled by varying the thickness of the materials and material parameters in order to study how the skyrmion phase will be changed. The structure and morphology, the transport properties, and the skyrmion phase of the grown samples will be investigated by means of X-ray diffraction, SQUID-VSM systems, and polarized neutron reflectometry experiment, respectively.

## The production and moderation of neutrons for a High Brilliance Neutron Source

ID 271

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*DOEGE, Paul (Forschungszentrum Jülich GmbH); BAGGEMANN, Johannes (Forschungszentrum Jülich GmbH); BÖHM, Sarah (NET, RWTH Aachen); CRONERT, Tobias (Forschungszentrum Jülich GmbH); VOIGT, Jörg (Forschungszentrum Jülich); RÜCKER, Ulrich; MAUERHOFER, Eric (Forschungszentrum Jülich GmbH); GUTBERLET, Thomas (Forschungszentrum Jülich); BRÜCKEL, Thomas (Forschungszentrum Jülich GmbH)*

Compact accelerator based neutron sources (CANS) offer the possibility to establish a network of neutrons sources to cover various activities in neutron research throughout Europe. Recent developments show that these sources are scalable, highly flexible, efficient and can be built and operated with reasonable costs as described in the High Brilliance neutron Source project (HBS) [1]. At such a source, neutrons are produced by protons or deuterons in the low MeV range impinging on a suitable target material. An optimized thermal and cryogenic moderator surrounding the target, moderates the neutrons to energies required by the instruments. This target / moderator assembly is a crucial part of a CANS defining the performance but also raising engineering challenges. The target has to withstand an average power of 100 kW and a cryogenic finger moderator needs to be operated in a narrow extraction channel. Additionally to these challenges the whole assembly has to be optimized to the needs of the instruments ranging from thermal neutrons with a short neutron pulse to long wavelength instruments with a long neutron pulse. We will present different target / moderator concepts for various instrument requirements built at a compact accelerator based neutron source.

[1] U. Rücker, et al.; The Jülich high-brilliance neutron source project; The European Physical Journal Plus 131, 19 (2016)

## 6Mg(NH<sub>2</sub>)/9LiH/LiBH<sub>4</sub>: Molecular Dynamics and Hydrogen Diffusion

ID 276

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There are several methods to store hydrogen, e.g. with physical technologies (compression), in metal hydrides or in complex hydrides. Within the complex hydrides, the metal amides Mg(NH<sub>2</sub>)<sub>2</sub>-LiH have recently gained in importance. They have good hydrogen storage properties with high capacity (ca. 4 wt%) and good reversibility. The drawback of these materials is the high kinetic barrier for hydrogenation, which results in a long loading time. This can be overcome with the addition of LiBH<sub>4</sub>, thus, the reaction is faster. So far Mg(NH<sub>2</sub>)<sub>2</sub>-LiH-LiBH<sub>4</sub> is an outstanding candidate to be used for hydrogen storage. Interestingly, some compositions can even reach operation temperatures below 100 degrees C and the desorbed products contain amongst others liquid phases. The hydrogen uptake and release properties of this system are well characterized in terms of capacity, kinetics and phase composition, but the basic mechanism how the reaction takes place and why it has these excellent storage facilities is still unknown. With 6Mg(NH<sub>2</sub>)<sub>2</sub>/9LiH/LiBH<sub>4</sub> we want to understand the system and the reaction mechanism. We will present first data of neutron scattering experiment using quasielastic scattering at TOFTOF to study hydrogen diffusion of the desorbed and absorbed state, as well as small angle neutron scattering to study particle sizes to gain further insight on the hydrogenation/dehydrogenation mechanism.

## Development of picosecond time-resolved nuclear resonance scattering and a novel synchrotron Mössbauer source at PETRA III

ID 278

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Nuclear inelastic scattering (NIS) is not bound by optical selection rules, which restrict the accessible vibrational modes in other techniques such as Raman scattering. It gives the phonon density of states (pDOS) at the instant the x-ray photon gets absorbed. Therefore, it is well suited to study the time evolution of pDOS in optically excited materials containing Mössbauer-active nuclei [1, 2]. We developed a time-resolved optical laser pump-NIS probe experiment at the beamline P01, PETRA III, DESY (Hamburg). 14.4 keV synchrotron radiation (SR) pulses formed the probe. Optical excitation was caused by time-delayed 250 fs laser (LR) pulses from a 1030 nm source, which was synchronized to the synchrotron ring and emits pulses at every 2nd or 4th SR pulse. We measure the dynamics of the excited state with a time-resolution of 100 ps by performing NIS of the SR pulse accompanied by the LR pulses. The ground state was measured in identical conditions using the SR pulse(s) in the absence of the LR pulse. We show the evolution of pDOS after laser excitation in a 3  $\mu\text{m}$  thick Fe foil and spin crossover complexes:  $[\text{Fe}(\text{PM-BiA})_2(\text{NCS})_2]$  and  $[\text{Fe}(\text{Htrz})_4(\text{trz})_2]$  (BF4). We also report the progress in the development of a novel synchrotron Mössbauer source based on the nuclear Bragg reflection from a  $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$  crystal.

[1] S. Sakshath et al, *Hyperfine Interact.* 238, 89 (2017)

[2] R. Röhlsberger, *Nuclear condensed matter physics with synchrotron radiation*, Springer (2004)

## Heavy ion-induced gas desorption in accelerators

ID 283

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Pressure increases in vacuum systems of particle accelerators have been observed since almost 50 years. In recent years, the dynamic vacuum turned out to be an intensity limitation in heavy-ion accelerators such as the heavy ion synchrotron SIS18 at GSI. Charge exchanged lost beam ions stimulate the release of gas from the chamber walls and the subsequent pressure increase leads to more beam-loss. Hence, the effect can lead to severe deterioration of the vacuum to the point of complete beam loss and is an issue for new heavy ion accelerators such as the FAIR facility or Spiral2 with highest beam intensities. To come up against the dynamic vacuum, several measures have been conducted to understand the physics behind the ion-induced desorption and to minimize the yields. The contribution summarizes the perceptions gathered to date, including pressure rise studies, materials analysis and modeling of the desorption process. Latest experiments on the annealing of critical components revealed the possibility to minimize the desorption yield of accelerator-relevant components by two orders of magnitude. The amount and composition of gas contained in materials was measured by thermal desorption spectroscopy and gives insight into the origin of desorbed gas for room temperature systems. At cryogenic systems, gas can be accumulated at the surface over time. Therefore, investigations on desorption of frozen gas ice layers were started.

## Instrumentation for compact High Brilliance Neutron Sources

ID 284

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The High Brilliance Neutron Source (HBS) concept [1,2] offers the optimization of the neutron spectrum and the time structure of the neutron pulse for the individual beam ports due to specific moderator configurations and dedicated target stations with suitable pulse structure. The HBS principle is scalable and can be realized at different power levels with well adapted instruments. In this presentation, we present some workhorse instrument concepts for neutron scattering as well as neutron analytics that can be realized at a low-power, university size neutron source of the NOVA ERA type [2]. In addition, we present a suite of instrumentation for elastic and inelastic neutron scattering and neutron analytics that can be realised at a high-power HBS source [1] that is scientifically competitive with the instrumentation at today's medium flux research reactors. We show instrument layout concepts together with flux and detector intensity calculations based on the moderator calculations for the neutron sources of the NOVA ERA and the high-power HBS type.

[1] U. Rücker et al., The Jülich high-brilliance neutron source project, Eur. Phys. J. Plus (2016) 131: 19. <https://doi.org/10.1140/epjp/i2016-16019-5>

[2] E. Mauerhofer et al., Conceptual Design Report NOVA ERA (Neutrons Obtained Via Accelerator for Education and Research Activities) A Jülich High Brilliance Neutron Source project, Schriften des Forschungszentrums Jülich, General, Volume 7, ISBN 978-3-95806-280-1 (2017) <http://hdl.handle.net/2128/16404>

## In-situ neutron diffraction and multi-scale modeling of mechanical behavior of the CNT/Al composite

ID 287

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Aluminum matrix composites (AMCs) today are widely used in automobile, aerospace and other engineering industries. Single-walled or multi-walled carbon nanotubes (CNTs) are attractive reinforcements for fabricating high performance AMCs, because of their extremely high strength and modulus, low density and good physical properties. So far, many works have shown that the CNTs can improve the strength and the stiffness of the composites. However, they can have adverse influences on the ductile failure properties like ductility and fracture toughness. In many literatures, the strengthening mechanisms of CNT/Al composites have been analyzed via analytical method [1-3]. However, in these analyses, the experimental evidences for the Orowan and the thermal mismatch strengthening mechanisms were not well established. Meanwhile, the analytical method cannot give deep insight into the micro-mechanism of strengthening of CNTs and deformation behavior of CNT/Al composites. In this study, a CNT/Al composite and a corresponding unreinforced Al alloy were manufactured via friction stir processing and rolling techniques. The in-situ neutron diffraction (performed at FRM II) during tension of the CNT/Al composite and the Al alloy was conducted. The crystal lattice strains were determined. A crystal plasticity finite element model was also developed to quantitatively predict the micro-mechanical behaviors of the composite and the alloy. The micromechanism of strengthening of CNTs and deformation behavior of CNT/Al composite were analyzed and discussed.

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[2] Bakshi SR, Agarwal A. *Carbon* 2011;49:533.

[3] Liu ZY, Xiao BL, Wang WG, Ma ZY. *Carbon* 2014;69:264.



## How does strain influence the performance in polycrystalline solar cells at the nanoscale?

ID 300

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Polycrystalline solar cells offer numerous advantages over their single crystalline counterparts, including lower cost, higher flexibility, and ease of fabrication. Unfortunately, polycrystalline cells are less efficient at converting sunlight into electrical power. Grain boundaries (GBs) are generally accepted as the cause of this efficiency differential because they act as recombination centers and induce voltage inhomogeneities. While GBs have been explored, the role of strain and its impact on local efficiency remains a potential avenue for improving efficiency in polycrystalline thin films. For this study, we have utilized the hard X-ray nanoprobe (HXN) at NSLS II to measure simultaneously and operando the performance, elemental distribution, and strain via X-ray beam induced current (XBIC) and voltage (XBIV), X-ray fluorescence, and X-ray diffraction. We find a positive correlation between the local strain and X-ray beam induced current and voltage in industrially manufactured Cu(In,Ga)Se<sub>2</sub> grains in a correlative microscopy approach. In grains with low angle tilt boundaries, the connection between strain and XBIC is unclear, likely due to the confounding influence of low angle GBs. Our correlative microscopy approach opens a new avenue for exploring the impact of strain, defects, and morphology on solar cell performance at the individual grain level and points to strain engineering as a potential direction for improving solar cell efficiency.

## In-situ light scattering at neutron beam lines - experiences made and challenges ahead

ID 304

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What is often well established at many synchrotron beam lines is still in the development phase at neutron beam lines: In-situ light scattering techniques. The science case for in-situ light scattering at neutron instruments lies mostly in the limited reproducibility of sample preparation and stability of the samples over time. Whereas many soft matter or hard matter samples are not transparent for light, many biological samples often show a sufficiently broad spectral range where light absorption does not play a dominant role. Natural candidates for neutron instruments to be equipped with in-situ light scattering techniques are small angle scattering, spin echo, time-offlight and backscattering beam lines. We routinely supply in-situ dynamic light scattering with one fixed scattering angle at the instrument KWS-2 at MLZ to users who would like to control their sample during the neutron measurement. Recently, we have successfully tested a three angle dynamic light scattering set-up at KWS-2. For the Jülich neutron spin echo spectrometer we are currently developing a prototype sample environment which includes two scattering angles and a transmission detector. The transmission detector reports on a change in turbidity with a very high time resolution. This is especially attractive to thermoresponsive soft matter samples with a very narrow transition from a swollen to a more compact micellar state.

**In-situ investigation of the sputter deposition of metal contacts on polymer thin films**

ID 308

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With their easy processability, high flexibility and tuneable optical properties, organic electronics offer a wide range of potential applications. Although based on organic materials, their electrodes are typically made from metals due to their unrivaled electronic conductivity. Thus, polymer-metal interfaces are inherently present and have a major influence on the device behavior. Understanding the growth mechanisms of metal contacts on polymer thin films plays a crucial role in identifying potential ways to enhance the device performance. We investigate the morphological changes during the sputter deposition of metal contacts onto photoactive polymer films via in-situ GISAXS [1]. This technique allows insights into the structural evolution of the metal on the organic film, which depends on various parameters such as the deposition rate or the film morphology. Making use of the brilliant synchrotron light source Petra III at DESY, we can study the film formation with outstanding spatial and temporal resolution. The metal layer formation can be described by applying an appropriate growth model based on our earlier work. [2] Comparing the deposition behavior of typical electrode materials on thin films of photoactive organic materials helps to understand their influence on the respective device performance.

[1] A. Hexemer, P. Müller-Buschbaum, IUCrJ 2, 106-125 (2015)

[2] M. Schwartzkopf et al., ACS Appl. Mater. Interfaces, 7(24), 13547-13556 (2015)

[3] M. Schwartzkopf et al.; ACS Appl. Mater. Interfaces, 9, 5629–5637 (2017)

## High-throughput X-ray microtomography for 3D digitization of insects

ID 314

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Synchrotron-based X-ray imaging has become an established method for the examination of small animals and X-ray microtomography in particular is an important tool for non-destructive 3D imaging of insects. The UFO imaging station of the KIT Synchrotron Radiation Facility is optimized for fast X-ray imaging and includes a high-speed detector system and a robot to change samples. The setup facilitates high-throughput experiments and 3D digitization of large numbers of insects in a single experiment. Despite faster acquisition times, image analysis is often still challenging and time-consuming. In particular, manual segmentation of tomographic volumes is extensive and the results often show unwanted artifacts. By employing semi-automated tools, which were developed in the scope of the interdisciplinary ASTOR and NOVA projects, image analysis can be accelerated and improved. Recent experiments demonstrate the value of fast X-ray imaging for the digitization and 3D analysis of both extant and fossil insects. Interactive 3D reconstructions based on tomography data allowed analyzing motion systems and the elytra of beetles proved to be interesting role models for biomimetic design. When studying mineralized insects from the fissure fillings of the Quercy region in France, we found that also insects from non-amber collections might contain well-preserved morphological characters, allowing species descriptions and phylogenetic analyses as done for extant specimens. A first study revealed the detailed anatomy of a hister beetle inside a stone matrix. In the following, we examined more than 1,500 mineralized fly pupae from the same locality and identified exceptionally preserved parasitoid wasps, providing both morphological and ecological data.

## Dynamics of proteins in aqueous solutions - recent advances using high-resolution neutron spectroscopy

ID 315

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Novel high resolution neutron spectrometers permit new experiments probing several hierarchically superimposed levels of protein dynamics on the molecular level, and the nanosecond time scale internal motion of proteins in aqueous solutions at low concentrations of 50 to 200 mg/ml can be studied [1,2]. These novel experiments access the in-solution dynamics of proteins and its difference to the previously studied hydrated macromolecular powder dynamics. Simultaneously, the superimposed global center-of-mass motions can be accessed and decomposed in the rotational and translational diffusion contributions [3]. Neutron backscattering permits a unique access to the short-time self-diffusion of proteins and other macromolecules in aqueous solutions [4]. On this time scale, hydrodynamic interactions govern the diffusive motion. Current topics are being explored, amongst others, of macromolecular crowding [4], of “patchy” colloid physics [5,6], of protein cluster formation [2,5], of protein unfolding [7,8], and of intrinsically disordered proteins [9].

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- [2] M.K.Braun et al., JPCL 8, 2590 (2017)
- [3] M.Grimaldo et al., EPJ Web of Conf. 83, 02005 (2015)
- [4] F.Roosen-Runge et al., PNAS 108, 11815 (2011)
- [5] M.Grimaldo et al., JPCL 6, 2577 (2015)
- [6] F.Roosen-Runge et al., Sci.Rep. 4, 7016 (2014)
- [7] M.Grimaldo et al., PCCP 17, 4645 (2015)
- [8] M.Hennig et al., Soft Matter 8, 1404 (2012)
- [9] S.Lenton et al., Biophys.J. 112, 1586 (2017)



## Investigating ion pairing in a liquid by electron-electron coincidence spectroscopy

ID 321

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The formation of ion pairs in an electrolyte solution has been investigated by numerous methods, arguably neutron scattering being one of the most powerful. Nevertheless, obtaining unambiguous results has been elusive for a lot of systems. We argue that non-local autoionization processes, in particular the so-called Electron Transfer Mediated Decay (ETMD), have the potential to become a probe for ion pairing that is based on a clearly different mechanism than existing techniques. In ETMD, an inner-valence or shallow core-level vacancy, typically created via photoionization with synchrotron radiation, is filled by an electron from the first solvation shell of the excited site. Using the excess energy of that process, another solvation shell electron is released into the continuum. The kinetic energy spectrum of that second electron, the 'ETMD electron', carries a fingerprint of the surroundings of the excited site. We have developed a set-up to record these ETMD spectra by electron-electron coincidence spectroscopy on a liquid jet. The coincidence technique removes the substantial background of inelastically scattered secondary electrons. Results are presented for solutions of Li and Mg-salts.

## Macromolecular Neutron Diffraction at the FRM II Neutron Source

ID 322

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Neutron single crystal diffraction provides an experimental method for the direct location of hydrogen and deuterium atoms in macromolecules, thus providing important complementary information to that gained by X-ray crystallography. At the FRM II neutron source in Garching near Munich the single crystal diffractometer BIODIFF, a joint project of the Forschungszentrum Jülich and the FRM II, is dedicated to structure determination of biological macromolecules. Typical scientific questions address the determination of protonation states of amino acid side chains, the orientation of individual water molecules and the characterization of the hydrogen bonding network between the protein active centre and an inhibitor or substrate. This knowledge is often crucial towards understanding the specific function and behaviour of a protein. BIODIFF is designed as a monochromatic diffractometer and is able to operate in the wavelength range of 2.4 Å to about 5.6 Å. This allows to adapt the wavelength to the size of the unit cell of the sample crystal. Data collection at cryogenic temperatures is possible, allowing studies of cryo-trapped enzymatic intermediates. Some recent examples will be presented to illustrate the potential of neutron macromolecular crystallography.

## The new Quenching & Deformation Dilatometer for Materials Science with Neutron Scattering at MLZ

ID 323

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A Quenching & Deformation Dilatometer (TA instruments DIL 805A/D/T) is now operational at the Heinz Maier-Leibnitz Zentrum (MLZ). It is customized for running neutron scattering measurements during temperature/deformation treatment of the sample, in particular neutron diffraction (phase, texture, stress/strain) at STRESS-SPEC and neutron small-angle scattering (nanostructure) at SANS-1. The dilatometer offers a simultaneous high-precision measurement of the length changes of bulk samples at all times, also when heating/cooling or deforming the sample, adding an additional measurement quantity that is sensitive to transformations in the sample. The combination of the neutron and dilatometry measurements yields a unique view on the microstructural evolution under thermomechanical treatment. The sample can be inductively heated and gas cooled according to a user-defined linear or exponential cooling rate. The temperature range is currently from room temperature up to 1500°C. The heating rate can be up to 4000°C/s, while specimens can be deformed with deformation rates between 0.01 and 200 mm/s. Depending on user demand, the temperature range can be extended down to -160°C with an additional furnace configuration. In addition to the dilatometer, we will present also first in-situ neutron scattering experiments.

## The new end-station PEAXIS for RIXS and XPS measurements at the BESSY II synchrotron

ID 324

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The electronic states and their dynamics determine important functional material properties such as charge and, via electron-phonon coupling, also heat transport. Thus a profound understanding of the microscopic mechanisms underlying electrical conductivity and its coupling to thermal conductivity is gained by probing the electronic structure. The new end-station PEAXIS built at BESSY II combines two important experimental methods for electron spectroscopy in a single UHV system. Resonant Inelastic X-ray Scattering (RIXS) and X-ray Photoelectron Spectroscopy (XPS) allow wavevector-resolved band mapping of electronic states in a broad range of functional materials. A dedicated focus is on solid state samples since PEAXIS features a large wavevector range accessible by the continuous rotation of the RIXS arm and offers solid-state sample manipulators covering a temperature range from 10 to 1000 K. In our presentation we report on the capabilities of the PEAXIS instrument and the results of our commissioning experiments. The beamline covers an incident energy range from 200 eV to 1200 eV and provides a beam spot size as small as  $3.8\mu\text{m} \times 12.4\mu\text{m}$  at the sample position. PEAXIS provides excellent energy resolution which has been experimentally demonstrated to exceed a resolving power of 10000. RIXS measurements on liquid acetone and on electronic d-states in NiO show that PEAXIS is competitive to state-of-the art RIXS instrumentation at third-generation synchrotron facilities.

## Ion-induced surface patterning and its application in nanofabrication via templated growth

ID 326

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*MALSCH, Gerald (Technische Universität Dresden); DE SCHULTZ, Ricardo (Technische Universität Dresden);  
FACSKO, Stefan (Helmholtz-Zentrum Dresden - Rossendorf)*

Low-energy ion irradiation of surfaces can lead to nanoscale pattern formation with a wide variety of morphologies, resulting from a number of interacting ballistic and diffusive mechanisms which govern the mass redistribution under ion irradiation. The choice of process parameters such as sample temperature or ion incidence angle determines the relative influence of these mechanisms and thereby the pattern morphology. After briefly outlining the patterning mechanisms and discussing the resulting morphologies on semiconductor surfaces, we present our approaches at templated nanostructure growth based on these ion-induced surface patterns. They include epitaxial nanowires via geometric shading, longrange chemical ordering in diblock-copolymer thin films, and engineering of magnetic anisotropy in topographically modulated thin films. The required technologies of low-energy ion irradiation, polymer chemistry, and physical vapor deposition are well-established and can readily be implemented at industrially relevant scales. Thus, nanostructured materials fabricated in such bottom-up manner have the potential to make substantial contributions to solving our society's present challenges: They can increase the sensitivity of diagnostical tools in medicine, lead to novel information technology, or enhance the efficiency of energy harvesting from renewable sources.



## Larmor diffraction -status and new concepts

ID 327

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*HABICHT, Klaus (Helmholtz-Zentrum Berlin); KEIMER, Bernhard (MPI for Solid State Research)*

We review the current status of Larmor diffraction (LD) at TRISP at the MLZ. The current setup with precession fields both up- and downstream the sample has a good resolution and is not sensitive to the crystal size or beam divergence. The disadvantage is that it is not possible to work with spindepolarizing ferromagnetic samples or with magnetic fields at the sample. There are two proposals for a LD configuration using precession fields only along the incident beam upstream the sample, such that spin-depolarization at the sample is not harmful. The first of these new concepts is the single-arm LD proposed by Rekveldt and van Well, the second is a spin modulation technique using one radio-frequency spin-flipper (Habicht, unpublished). Both new concepts loose resolution in comparison to the classical LD. We will discuss and compare these new LD methods.

## T-REX, a bi-spectral chopper spectrometer for the ESS

ID 333

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T-REX is a bispectral direct-geometry chopper spectrometer accepted for construction at the European Spallation Source (ESS). Currently the project is in the engineering design phase. We will discuss technical solutions for various instrument components. The instrument will provide neutrons in the energy range  $2 \text{ meV} < E_i < 160 \text{ meV}$  and therefore will allow detailed exploration of reciprocal space to study collective excitations of the nuclear or spin structure as well as localized excitations. The chopper layout [1] is designed for polychromatic experiments using multiple incident neutron energy in Repetition Rate Multiplication. Moreover, the chopper configuration can be tuned to high energy resolution, i.e. achieving 20 micro-eV elastic energy resolution at 2 meV incident energy, or to relaxing the resolution and gain up to 5 times greater flux at the sample. By limiting the beam collimation, the Q resolution can be tailored to the experimental needs, as investigated in the study of the instrumental resolution function [2]. Many of the scientific questions to be addressed by the spectrometer will be tackled by means of neutron Polarization Analysis (PA) to probe not only the energy of spin excitations but also the eigenstates or to distinguish the spin-incoherent from the coherent scattering. T-REX features XYZ neutron spin polarization analysis. Neutron polarization is achieved with the SEOP setup [3]. Neutron spin analysis in XYZ is performed with the magic PASTIS setup [4]. Specific investigations of high quality  $^3\text{He}$  cells are required [5], to achieve a long decay time of the polarization of the gas, which is crucial for long lasting inelastic scattering experiments. In collaboration with the ESS detector group we are designing the PSD detectors based on the 10B multigrid technology [6] to cover the large area ( $20 \text{ m}^2$  in the full scope) with minimal gaps and a high detection efficiency for neutrons of energy up to 200 meV.

[1] J. Voigt, N. Violini, T. Brückel, NIMA 741, 26 - 32 (2014)

[2] N. Violini, J. Voigt, S. Pasini, T. Brückel, NIMA 736, 31 - 39 (2014)

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[4] E. Babcock, Z. Salhi, P. Pistel, A. Ioffe, G. Simeoni, J. of Phys / Conf. Series 528, 012018 (2014)

[5] Z. Salhi, E. Babcock, A. Ioffe, P. Pistel, J. of Phys / Conf. Series 528, 012015 (2014)

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## Crystal structures and phase transitions of inorganic-organic hybrid layered materials (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>3</sub>)<sub>2</sub>MCl<sub>4</sub> (M = Cu<sup>2+</sup> and Mn<sup>2+</sup>)

ID 336

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Because of the combination of the hydrogen bonds between halogen atoms in an inorganic part and nitrogen atoms in an organic part, a layered inorganic-organic hybrid perovskite (C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>3</sub>)<sub>2</sub>MeCl<sub>4</sub> (Me = Cu<sup>2+</sup>, Mn<sup>2+</sup>, Fe<sup>2+</sup>) shows a various crystal structures [1, 2, 3]. To understand an interlayer spacing effect on a magnetic property, various systems have been developed. All three Cu-, Mn and Fe-compounds crystallized in the same orthorhombic space group (Pbca, No. 61) at room temperature (a = 7.207Å, b = 7.301Å, c = 39.413Å for Mn-compound, a = 7.302Å, b = 7.327Å, c = 38.635Å for Cu-compound, and a = 7.171Å, b = 7.279Å, c = 39.106Å for Fe-compound, respectively). However, in the recent study [1], it is reported that Cu-compound should be ferroelectric, although the inversion center in the point group mmm prevents the spontaneous polarization. In addition, it is also claimed that Mn-compound should have an additional phase transition at 100K and below 100K the point group of Mn-compound is mm2 (Pbc21), where the ferroelectric property is allowed [4]. But from our own investigations on Mn- and Cu-compounds [2, 5] and also a recent study on Fe-compound [3] strongly suggest that Cu-, Mn- and Fe-compounds are not ferroelectric at room temperature but ferroelastic. In this study, we will show crystal structures of Mn- and Cu-compounds not only from neutron single crystal diffraction techniques but also other methods including temperature dependent X-ray powder diffraction results and domain structures under polarized microscope.

- [1] A. O. Polyakov et al., Chem. Mater., 24 (2012) 133.
- [2] S. H. Park et al., Dalton Trans., 41 (2012) 1237.
- [3] Y. Nakayama et al., Angew. Chem. Int. Ed. 56 (2017) 9367.
- [4] A. Polyakov, (2015) Ph.D Thesis, University of Groningen.
- [5] G. Park et al., (2018) in preparation.



## The Materials Science group at Heinz Maier-Leibnitz Zentrum (MLZ)

ID 337

*GILLES, Ralph*

The Materials Science group consists of more than 30 people working in a variety of fields related to the applied materials science. Members of this group belong to neutron scattering or positron spectroscopy instruments including the staff acquired through 3rd party funding and the group of fuel cell development. Each month a group meeting is organized to exchange the activities of the group members, especially their scientific work. In the meetings short presentations are given by group members to introduce the methods and the scientific topics of their studies. Typical tools applied in the group are diffraction, small-angle scattering, prompt gamma activation analysis, radiography/tomography, inelastic scattering with time of flight method and neutron depth profiling. Besides development for neutron scattering instrumentation (neutron depth profiling at PGGGA instrument, implementation of a testing machine for Stress-Spec and SANS-1 instrument, positron beam experiments, radiography and spectroscopy instruments at ESS) the topics of our scientific studies are: high performance alloys, energy related materials (batteries, hydrogen storage), electronic structure of correlated materials, Fundamental properties of plasmas, archeological objects and last but not least the development of a future MEU fuel element for FRM II.



## Phase behavior and solution structure of new UCST and LCST-type polymers

ID 339

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Thermoresponsive polymers are an interesting class of material for industrial and medical applications and also for fundamental research. Lower critical solution temperature (LCST) polymers, such as poly(N -isopropylacrylamide), are common and well-studied, whereas materials with an upper critical solution temperature (UCST) still are rare and not well examined. Depending on the material used, the transition temperature can be varied by different parameters, which can enlarge the potential field of applications. Here, not only it is interesting to tailor the temperature, at which the phase transition occurs, also it is interesting to control the type of transition, i.e. from LCST to UCST or vice versa. We synthesized acryl-amide based copolymers that, depending on the composition, display either a LCST behavior, or a UCST-like phase behavior. We used turbidity measurements, static and dynamic light scattering as well as small angle neutron scattering (SANS) to study the phase behavior of our polymers on global and local length scales. In particular, from SANS important information on the polymer structure is accessible.



## Structure and Dynamics Polyelectrolyte/Microemulsion Complexes (PEMECs) Studied by Neutron Scattering

ID 341

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Mixtures of oppositely charged polyelectrolytes (PE) with oppositely charged microemulsion (ME) droplets were studied with respect to their phase behavior and the structures present in solution. In this work we varied the size of the ME droplets, their charge density, the mixing ratio, ionic strength of the solution, and the type and Mw of the PE. Their structural properties were characterized by means of static and dynamic light scattering, zeta-potential measurements, and as main methods by small angle neutron scattering (SANS) and neutron spin-echo (NSE). In the phase behavior a systematic dependence on the Mw of the polyelectrolyte was observed. Depending on the mixing ratio different elongated structures of connected droplets are observed, whose elongation depends on the Mw and choice of the polyelectrolyte. The interconnected nature of the droplet aggregates leads to changes in dynamics which were studied using pulsed field gradient NMR (PFGNMR), neutron spin-echo (NSE) spectroscopy and fluorescence correlation spectroscopy (FCS), thereby yielding detailed insights into the dynamic aspect of such mixed colloidal systems. These experiments give a comprehensive picture of the formed complexes and allow for a detailed understanding of the interactions between charged ME droplets and PEs. With their high solubilization capacity they are interesting for potential applications in formulations, which require optimized delivery.



## Complementary neutron/synchrotron study of structure and magnetism of rare earth/transition metal multilayers

ID 343

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Magnetic nanostructures containing thin layers of ferromagnetic and nonmagnetic metals are of considerable interest both for fundamental physics and as potential elements for spintronic devices. Exchange coupling of magnetic nanolayers separated by paramagnetic spacers is relatively well studied for purely rare earth (RE) systems or purely transition metal (TM) ones but little is known about interlayer exchange coupling RM and TM magnetic metals through paramagnets. In this work we report results of neutron/X-ray combined study of several RE/TM systems as Fe/Pd/Gd, Dy/Co, Dy/Gd etc. Systems were grown with UHV sputtering, good layered structure was confirmed with X-ray reflectometry. By combining neutron and resonant X-ray reflectometry we found rich phase diagram of RE/TM heterostructures which arising due to the competition of exchange coupling, magneto crystalline anisotropy and Zeeman energy. The research was partly supported by RFBR under Grant No. 18-32-00197

## Neutron activation analysis (NAA) reveals “fingerprint” of materials: From microchips to meteorites

ID 348

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With its very pure thermal neutron flux, the reactor FRM II at Garching offers very good opportunities for studies of chemical composition in samples. The advantages of the instrumental NAA (INAA) are simple sample preparation without chemical handlings, high sensitivity, multi-element capability at all concentration levels (main, minor & trace) and almost non-destructiveness. The so-called “fingerprint” method, especially analysing rare earth elements (REEs), can give us e.g. more details of the provenance of the samples. INAA is applied in material science e.g. quality assurance of semi-conductor materials. Impurities in the sub-ppb-level (1:10<sup>9</sup>) can be detected after long-time irradiation with high neutron flux (> 10<sup>14</sup> cm<sup>-2</sup>s<sup>-1</sup>). One of the interesting interdisciplinary projects is the bulk analysis of meteorites. Among them is the stony meteorite “Cloppenburg”, which was found in 2017 as the 49th German meteorite [1]. More than 45 elements could be determined by INAA [2]. The INAA data is mandatory for the interpretation of cosmogenic radionuclide data determined by accelerator mass spectrometry (AMS) [3] to reconstruct the history of the meteorite(s) such as irradiation and terrestrial age, and preatmospheric size.

[1] Meteoritical Bulletin, no. 106, in prep. (2018).

[2] X. Li et al., Proc. of Paneth-Kolloquium (2017).

[3] G. Rugel et al., this meeting.

## PtyNAMI: Ptychographic Nano-Analytical Microscope at PETRA III -How to achieve sample stability in the nanometer range

ID 349

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In recent years, ptychography has been established as a method in X-ray microscopy to achieve a spatial resolution even below the diffraction limit of x-ray optics, down to a few nm. This requires, among other things, an extremely high degree of mechanical stability, a low background signal from the x-ray microscope and highest demands on the beam guiding and focusing optics. PtyNAMI is the new generation hard x-ray scanning microscope at beamline P06 of PETRA III at DESY combining a sample scanner designed for maximal stability, a new detector system designed to reduce background signals, and an interferometric position control of sample and X-ray optics. The interferometer system enables tracking the sample position relative to the optics in scanning microscopy and tomography on all relevant time scales. This is crucial for high-resolution scanning x-ray microscopy to track vibrations and long-term drifts in the noisy environment of a synchrotron radiation source in user operation. We present the design concept in detail with a special focus on real-time metrology of the sample position during 3D x-ray scanning microscopy using a ball-lens retroreflector.

## Potential of in situ triaxial stress experiments using neutron time-of-flight diffraction on porous rocks for reducing the risk of subsidence and induced seismicity through gas production

ID 350

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In gas production fields the extraction of massive gas volumes from the geological underground caused subsidence up to about 30 cm and increased the regional seismicity. We consider the geomechanical issues of the increased seismicity due to hydrocarbon production, especially the coupling of reservoir pore pressure and in situ stress. Models cannot consider reservoirs where stress changes and pore pressure changes are decoupled from one another: Observations in numerous reservoirs show that the rate of minimum horizontal stress change is coupled to approximately 50-80% of the rate of pore pressure depletion due to gas production. This is critically dependent on the effective Biot stress coefficient. It can be derived from the coefficient of the dry bulk modulus of the rock and the bulk modulus of the mineral grains of the rock. A triaxial pressure cell with the possibility to apply axial, confining and pore pressures equivalent to those of hydrocarbon or geothermal reservoirs has been developed and manufactured were used for in situ stress experiments at the neutron time-of-flight stress/strain instrument EPSILON at beamline 7A of the pulsed neutron source IBR-2M, JINR Dubna for studying this rock behaviour

## Structural properties and room temperature ferromagnetism in nanocrystalline HfO<sub>2</sub>

ID 352

**KUMAR, Sandeep** (*Indian Institute of Technology (BHU)*)

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Hafnium oxide (HfO<sub>2</sub>) crystallizes in the monoclinic phase at room temperature which undergoes a phase transformation to tetragonal and cubic at 1700 °C and 2600 °C, respectively. Herein, we present a detailed structural and magnetic properties of nanocrystalline HfO<sub>2</sub> powders synthesized by a Pechini type sol-gel method followed by calcination at 900 °C in air. X-ray diffraction and Le-Bail profile refinement revealed that HfO<sub>2</sub> crystallized in monoclinic phase, space group, P2<sub>1</sub>/c with an average particle size of ~40 nm. The lattice spacing calculated from high resolution transmission electron microscopy (TEM) micrograph and selected area electron diffraction (SAED) pattern further endorse the formation of monoclinic phase in HfO<sub>2</sub>. Magnetic measurements have shown that HfO<sub>2</sub> exhibits a noticeable hysteresis loop manifesting ferromagnetism at room temperature in contrast to the diamagnetic behaviour in bulk. Room temperature ferromagnetism observed in nanocrystalline HfO<sub>2</sub> has been the subject of great interest for spintronic device application. However, the origin of intrinsic ferromagnetic ordering in diamagnetic systems like HfO<sub>2</sub> is still not clear and sought further systematic studies. In order to have a better insight, polarized neutron reflectometry (PNR) technique can be utilized efficiently for probing the magnetic properties of HfO<sub>2</sub> thin films. PNR provides feasibility of excluding direct magnetic contributions of substrates resulting in absolute magnetic behaviour analysis with high accuracy.



## Investigating interfaces and spinterfaces of organic radicals by X-ray based spectroscopies

ID 353

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*CASU, Maria Benedetta (Universität Tübingen)*

Using soft X-ray spectroscopies, performed also at synchrotrons, we investigate thin film processes, surfaces and interfaces at the nanoscale, when organic radicals are deposited on metal and metal oxide surfaces. We suggest how to design organic radicals bearing in mind the thermodynamic factors that govern thin film stability, with the purpose of obtaining not only a chemically stable radical, but also stable thin films. We investigate the thermal and air stability of the deposited films, and we explore the influence of the surface/radical chemical bond and the role of surface defects on the magnetic moment at the interface. Our work shows that the use of X-ray based techniques represents a powerful approach to reveal the mechanisms governing complex interfaces, such as radical/metal and radical/metal-oxide where it is important to describe both charge and spin behavior (spinterfaces). A deep understanding of stable radical/inorganic spinterfaces may open the way to use radicals in solid state devices, or as quantum bits with dedicated configurations, as proposed for other molecular quantum bits, and in spin-based electronics.

## Surface Modes in Phospholipid Membranes

ID 356

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Phospholipid membranes are the basic construction material of cell membranes. Also, solutions of phospholipid vesicles find a wide array of applications in technical, medical and biological applications. In our previous publications we showed both the structure and the dynamic behavior of L- $\alpha$ -phosphatidylcholine (SoyPC) phospholipid membranes. [1,2] We established a multi-lamellar structure as well as a surface mode, that we attributed to a standing wave in the membranes. Following up on this experiment, we performed additional GISANS measurements. We were able to find the coherent scattering contribution of the dynamic modes of the phospholipid membranes with GISANS resolution settings specifically adapted to those features. Moreover, as we assumed the standing wave was linked to a specific phase of the lipid membrane, we investigated the system at different temperatures and could show that the standing wave indeed disappeared at temperatures below 25 degrees C and reappeared when the system was reheated. After showing the observed standing mode in GINSES experiments is most pronounced at physiological temperatures and vanishes after decreasing the temperature below 25 degrees C we can speculate that this feature of phospholipid membranes is indeed part of the natural function of such membranes in biological systems.

[1] S. Jaksch, H. Frielinghaus et al, Phys. Rev. E 91(2), (2015) 022716.

[2] S. Jaksch, H. Frielinghaus et al, Scientific Reports 7(1), (2017) 4417.

## The Sample Environment Communication Protocol (SECoP)

ID 363

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The integration of sample environment equipment in a neutron or synchrotron experiment is a complex problem both in the physical world and in the digital world. Different experiment control softwares offer different interfaces for the connection of sample environment equipment. Up to now there exists no software standard for this task. Therefore it is time-consuming to integrate new sample environment or to share sample environment equipment between facilities. The Sample Environment Communication Protocol (SECoP) is intended to solve this problem offering a standardized communication protocol for the integration of sample environment equipment. The development of the SECoP is a collaborative effort within the framework of the International Society for Sample Environment (ISSE) additionally supported by the european SINE2020 project. The SECoP is defined in a way that it is compatible with a broad variety of soft- and hardware operated at the different neutron and X-ray facilities. The SECoP is easy to implement facilitating the integration of sample environment equipment developed by external users which may have limited programming experience. The adoption of this standard will greatly facilitate the installation of new equipment and the sharing of equipment between the facilities. Presently first implementations of SECoP are tested at different facilities. Supply companies have been contacted as well. In this presentation we will give an overview of the present status of SECoP.

## Lattice Dynamics of Epitaxial Strain-Free Interfaces

ID 364

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We studied the phonon properties of ultrathin Fe<sub>3</sub>Si layers in Ge/Fe<sub>3</sub>Si/GaAs heterostructures as a model system for epitaxial, strain-free interfaces. Epitaxial Fe<sub>3</sub>Si layers with thicknesses from 3ML to 36ML were grown on GaAs(001) substrates and capped by a 4 nm thick amorphous Ge layer. Sample characterization with various methods showed the formation of epitaxial Fe<sub>3</sub>Si nanostructures with perfect stoichiometry and high interface quality. Nuclear Inelastic Scattering was used to determine the iron-partial phonon density of states at room temperature as a function of layer thickness. The results exhibit up to a two-fold enhancement of the low-energy phonon states compared to the bulk material for layer thicknesses of 8 monolayers and below. First-principles calculations explain the observed effect by novel, interface-specific phonon modes originating from the significantly reduced atomic force constants and allow for achieving a comprehensive understanding of the lattice dynamics of epitaxial, strain-free interfaces. The work was financially supported by the Helmholtz Association (VHNG-625) and the BMBF (05K16VK4).

## High Resolution Neutron Detection using Gridpix chips

ID 368

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*SCHMIDT, Fabian (Physikalisches Institut, University of Bonn); GRUBER, Markus (Physikalisches Institut, University of Bonn); KAMINSKI, Jochen (Physikalisches Institut, University of Bonn); DESCH, Klaus (Physikalisches Institut, University of Bonn)*

The world of detectors used in thermal neutron scattering instrumentation has changed. By alerts on the future Helium-3 supply, critical to perspectives of the large-scale research infrastructures, the run on substitutional technologies started. Most of the solutions could be adapted from developments of particle physics and are comprised of one or more layers of Boron-10. The Time Projection Method achieves a very high resolution by projecting ionization tracks onto a highly granular readout. The University of Bonn develops a novel system based on reconstructing boron conversion tracks using the Timepix - CMOS based chips with 55 micrometer sized pixels operated at clock speeds up to 80 MHz. Each matrix of 256 x 256 pixels is equipped with an InGrid - microstructured aluminum meshes 50 micrometer on top of the pixels serving as a charge amplifier. In a first prototype with 8 Timepix chips, which are arranged in parallel to a boron layer, the track topology with this unrivaled high resolution has been studied. By reconstructing the origin of the conversion ions a time resolution of <math><50\text{ ns}</math> and a spatial resolution of 100 micrometer has been achieved. As this setup now allows the full reconstruction of the conversion tracks down to the electron level the understanding of the track topology can help interpreting the signals in more coarsely resolved systems. The aim of the project is to prove the feasibility of realizing a neutron Time Projection Chamber.



## Polymer membranes analyzed by Elastic Recoil Detection and Positron Annihilation Lifetime Spectroscopy

ID 372

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Extensive characterization is needed to understand the physicochemical properties of polymeric membranes that are used for water purification. Currently, most techniques characterize the (near)-surface region of the membrane, even though its bulk obviously also plays a significant role in the final membrane performance. To achieve depth-profiles of the elemental composition of both integrally skinned asymmetric (ISA) and thin-film composite (TFC) membranes, elastic recoil detection (ERD) is introduced to the field as a potentially highly valuable technique to complement for instance XPS, EDX or RBS. It also allows to analyse remnants from chlorination of nanofiltration (NF) and reverse osmosis (RO) membranes being of high importance for the water treatment industry. Volume-averaged chlorine-uptake as well as complete Cl- and H-profiles as a function of membrane depth were obtained after NaOCl cleaning procedures at high pressures (e.g. 10 bar for 2.5 h) of polyamide (PA)-based thin film composite (TFC) membranes. The decrease in Hcontent upon chlorine exposure could be quantitatively proven for the first time and the influence of pressure, pH and chlorine feed-concentration on the location of chlorine in the membrane was studied. More chlorine is present deeper in the PA-layer with increasing chlorine uptake, either by increasing chlorine dose or by decreasing pH. The chlorine uptake goes in-line with reduced positron lifetime indicating a reduction of open pore size. It demonstrates a “tightening” of the membranes with reduced water permeability.

## X-Ray Investigation of Structure and Kinetics of Photoswitchable Lipid Monolayers

ID 373

**WARIAS, Jonas**

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Having integrated Amphiphilic photoswitchable molecules into phospholipid membranes, we investigate their biologically relevant properties. By studying the interactions in such a model system consisting of switchable biomimetic molecules in lipid membranes here, we study the structure and kinetics of membrane response to the switching process. These in situ experiments not only contribute to the fundamental understanding of membrane dynamics but also will contribute to potential applications for molecular switches such as drug delivery. In order to investigate these properties we study model systems in which amphiphilic photoswitchable molecules are integrated into Langmuir films of phospholipids. We have modified glycolipids to contain an azobenzene photoswitch between the chain and the head group and successfully embedded those in a monolayer of Dipalmitoylphosphatidylcholine (DPPC). This allows us to reversibly change the azobenzene-glycolipid orientation between trans- and cis-conformation by illumination with UV and blue light. We have followed the structural changes in this model membrane and the switching kinetics of the system with Langmuir isotherms and in situ X-ray methods, X-ray reflectivity and grazing incidence diffraction. The membrane structure responds on a time scale of seconds upon UV illumination. Switching from trans to cis conformation results in changes in molecule tilt and spacing and also membrane thickness. A critical point in surface pressure at 18.4 mN/m has been discovered above which the membrane expands rather than compresses upon switching from trans- to cis- conformation.

## Pharmaceutical Nanoparticles – Study by Neutrons, Photons and DLS

ID 374

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Specific target Nanoparticles for therapy of cancer and other diseases were assembled from lipids, polymers and bio-ferrofluids. For cell targeting proteins were bound to the surface (corona). The structure and dynamics was investigated by SANS, DLS, SAXS and ASAXS as the multi-domain particles depict a size of 20-200 nm. Structure-material domains were localized by contrast variation in SANS and ASAXS. The structure of the modified nanoparticles is analyzed by dynamic light scattering DLS in combination with neutron small angle scattering SANS and metal specific X-ray scattering ASAXS. The multi-targeting modification is applied to drug and lanthanide loaded polymer nanoparticles (metal-PLGA, patent of the Gutenberg-University for RT), modified bio-polymers and liposomes as fast development system. The final targeting protein modification is followed by time resolved SANS with deuterium contrast variation in combination with dynamic light scattering (SANS-DLS). The bio-compatibility and the therapy effect of nano-drug constructs are proven in our time-resolved cell culture EPN test (exponential necrosis and proliferation test). Oral nanodrug application is tested with time-resolution by a simulator device of the gastro-intestinal tract with SANS-DLS observation.



## New Opportunities for Time-Resolved Photoelectron Spectroscopy at the European XFEL

ID 375

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A new SASE3 beamline branch at European XFEL in Schenefeld is currently being installed. It will be available to a large user community for time-resolved pump-probe photoelectron spectroscopy (TR-XPES) of solids and surfaces. SASE3 provides ultra-short (10-100 fs), extremely intense (1 - 2 mJ) pulses of coherent soft X-ray light (0.27 - 3.0 keV photon energy), which is ideally suited for comprehensively investigating matter on the atomic length scale with femtosecond temporal resolution: Spin- and time-resolved ARPES will allow tracking the dynamics of electronic structures on the fundamental time scale of electronic motion. Time-resolved XPS and XPD will allow monitoring the temporal evolution of chemical processes and structural dynamics, respectively, in real time. The X-ray beam can be varied in size at the sample position between 20 and 100  $\mu\text{m}$ , matching the typical focal size of electron spectrometers. Two dedicated experimental stations have been developed, a spin-resolving photoelectron momentum microscope (University of Mainz) and a time-of-flight spectrometer (University of Hamburg). The new branch, however, was designed flexible as a “multi-purpose” branch, allowing users to bring their own transportable experimental stations and operate them with the FEL beam. The branch is expected to become operational at end of 2019.

## In situ investigation of electrodeposition at liquid-mercury interfaces by X-ray reflectivity

ID 378

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Thanks to the high quality crystalline form of obtainable nanostructured material, due to the lack of substrate-induced stress or strain, and to the possibility of controlling growth parameters by applying an interface potential in electrodeposition, the interest in the study of growth processes at liquid-liquid interfaces has been lately renewed. With the aim of understanding nucleation and subsequent growth of crystals at Hg-electrolyte interfaces, experiments using a combination of electrodeposition, in situ XRR and time-resolved XRD were performed, employing the high resolution diffractometer LISA [4,5], at P08, PETRA III synchrotron, Hamburg. In previous studies, electrolyte containing 0.01M NaF+0.01M NaBr+0.5mM PbBr<sub>2</sub> was found to exhibit an adlayer growth [6,7]. Changing the potential from values < -0.8V vs Hg/Hg<sub>2</sub>SO<sub>4</sub>, where the Pb ions are amalgamated in the Hg, to values > 0.7V, these ions are deamalgamated, leading to the growth of a monolayer followed by 3D nanocrystal formation of PbBrF. Currently a fluoride free electrolyte is used: 0.01M NaBr+0.05mM PbBr<sub>2</sub>. While XRR curves show no evidence of growth at this concentration, experiments at higher concentrations (0.5mM PbBr<sub>2</sub>) clearly exhibit crystal growth. Also a different electrodeposition system was studied: Ge growth from 0.1M Na<sub>2</sub>SO<sub>4</sub>+0.05M GeO<sub>2</sub> electrolyte on Hg electrodes [1]. These investigations will aid understanding of the first stages of nucleation and growth by electrodeposition at liquid-liquid interfaces.

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- [3] J. DeMuth et al., Cryst. Growth Des, 2018, 2, 677-685
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## Rationally improving Pt-ceria based exhaust gas catalysts by time and space resolved operando QEXAFS

ID 381

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Converting pollutants like hydrocarbons or CO at low temperatures is a main challenge in exhaust gas aftertreatment. A promising approach for improving the low temperature activity of exhaust gas catalysts – typically supported noble metal nanoparticles – is to exploit the interaction between the noble metal component and support material like ceria (1). Recently, we demonstrated the rational tuning of the Pt particle size on ceria: Short reductive pulses allowed to increase the catalytic activity (2). Because of the highly dynamic nature of these catalysts, in situ characterization methods with a high time resolution are mandatory to identify and follow structural changes. Complementary quick scanning extended X-ray absorption fine structure spectroscopy (QEXAFS) measurements during reductive treatment at the Ce L3- and Pt L3-edges were used to elucidate the vivid interplay between Pt and CeO<sub>2</sub> (3). Furthermore, the activation treatment has been followed in a spatially and time resolved manner using simultaneously operando Pt L3 QEXAFS and infrared thermography to identify key parameters for tuning the Pt particles. The study reveals the dynamic behavior of Pt/CeO<sub>2</sub> based catalysts even at low temperatures and pinpoints to the importance of in situ characterization for rationally enhancing catalytic systems.

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## The New DynaMax endstation at the FemtoSpex Slicing facility for ultrafast dynamics

ID 386

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Ultrafast magnetization dynamics is a complex and fascinating field in fundamental physics and motivates studies on new magnetic recording techniques based on, e.g., all-optical switching (AOS) of magnetic order [1,2]. For our studies on photo-induced magnetization dynamics in rare-earth metals [3,4] the FemtoSpex slicing beamline at BESSY II is ideal since magnetization can be probed state- and element-specific with a time-resolution of 130 fs. In order to push the research on ultrafast dynamics to a new level the BMBF-financed DynaMax project was initiated. This project is a collaboration between the HZB, MBI and FU Berlin to build up a complete new endstation for the slicing beamline. We will present details of this innovative setup and new feasibilities for future studies. The operation will start in summer 2018 and our planned investigations will benefit from high magnetic fields with variable orientation and high ramping rates. The new endstation will further realize simultaneous measurement of magnetic dynamics in transmission, scattering and holography, back-pump front-probe geometry, THz experiments and in-situ sample preparation of complex magnetic systems with molecular beam epitaxy and sputter deposition.

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## peakR: An Open-Source Nonlinear Curve Fitting Package

ID 388

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peakR is an open-source package for nonlinear peak fitting that was developed using the popular statistical programming language, R. By utilising a modified Levenberg-Marquardt algorithm, the sum of both nonlinear and linear functions fitted to experimental data can be solved. The package consists of a set of R functions and an RStudio Addin that allows the user to analyse their dataset through either an interactive graphical user interface (GUI) or by using programming code to enable a smooth data analysis workflow irrespective of the user's analysis preference. As nonlinear peak fitting is a commonly used post-acquisition data analysis procedure in surface science, peakR specialises in analysing X-ray photoelectron spectroscopy (XPS) spectra. In particular, it enables chemical peak identification and labelling by comparing and matching experimental values to the built-in "Element Binding Energy" database.

## A Structural Investigation of Transition Metal Antimonates

ID 393

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Antimony based transition metal oxides have been researched and studied for years due to the interesting semiconductor and photocatalytic properties they exhibit. Whilst most transition metal antimonates (e.g.  $\text{NiSb}_2\text{O}_6$ ,  $\text{ZnSb}_2\text{O}_6$ ,  $\text{CoSb}_2\text{O}_6$  etc.) exist in a tetragonal trirutile structure,  $\text{CuSb}_2\text{O}_6$  exhibits a slight monoclinic distortion due to Jahn Teller effects dominating the structure. In our research, an investigation was undertaken to investigate how doping Ni into a  $\text{CuSb}_2\text{O}_6$  lattice would affect the structure of the material. It is well known that the structure of a material defines its properties, and through a systematic study we seek to understand this relationship within such antimonates. Both bulk powder and single crystal samples were synthesised for this work, using high temperature sintering and chemical vapour transport respectively. In the doped powder samples, XAS measurements indicate that some of the Cu ions are being reduced from  $\text{Cu}^{2+}$  to  $\text{Cu}^{1+}$  in the structure, reducing the effect of the Jahn Teller distortion and removing the monoclinic distortion. There has also been indication that there may be a potential low temperature phase transition occurring in the  $\text{NiSb}_2\text{O}_6$  structure, analysed through a single crystal diffraction across a decreasing temperature range.



## MLZ Science Group “Quantum Phenomena”

ID 396

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The MLZ Science Group “Quantum Phenomena” acts as a platform to promote and strengthen a close cooperation, in the fields of magnetism, superconductivity and strongly correlated electrons, for scientists at MLZ and from the surrounding institutions and universities. Together with the MLZ service and instrumentation groups, the Quantum Phenomena Group has played an important role in defining the MLZ roadmaps for science, sample environments and science-driven instrumentation. In this poster, an overview and the recent activities of the Quantum Phenomena Group will be presented. The new developments in neutron instrumentation and sample environments for the magnetism and quantum phenomena community will be given.

## Influence of Microstructure on Symmetry Determination of Piezoceramics

ID 399

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Frequently symmetry determination in studies is based on the discussion of agreement factors or the quality of the refinements, rather than on the basis of physical arguments. Especially in the field of piezoceramics this can be observed in the discussion whether monoclinic symmetry can be observed or not. In this study we could show with temperature-dependent high-resolution X-ray and neutron diffraction that based on agreement factors alone the physical origin of observations cannot be revealed. Only in combination with additional electron microscopy and electron probe microanalysis we could elucidate that a segregation of substituents results in a complex reflection splitting and phase coexistence that can be misinterpreted as monoclinic symmetry. This single-phase monoclinic Pm model is able to perfectly reproduce the diffraction patterns and is known from literature [1,2]. A model with phase coexistence of two classical orthorhombic Amm2 phases can reproduce the diffraction data with equal accuracy. This demonstrates the need of comprehensive analyses with complementary methods to cover a broad range of length scales as well as considering both average and local structure. The conclusions drawn from this work will have an impact on a broad range of research areas where inhomogeneities cannot be ruled out. The underlying mechanisms of the extraordinary properties of some functional materials originate not just in their structures but also their complex microstructures. Consequently, knowledge of both features of materials may be essential for the exploitation and development of their functionalities as well as improvement of material properties.

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## Dynamic response of high power target and beam dump materials to short pulse ion beam-induced pressure waves

ID 403

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Under extreme beam-induced temperature and pressure conditions at the planned FAIR facility, but also at HiLumi LHC and neutrino facilities, new candidate materials and design solutions have to be found to overcome the increased radiation dose and energy density effects in components that see directly the primary beam such as targets, beam windows, beam catchers and beam dumps. The dynamic response to pressure waves induced by short intense ion beam pulses for different graphite grades, as well as high damping carbon materials and carbon-carbon composites, was studied using 1 GeV U ions at UNILAC accelerator at GSI and 440 GeV protons at the HiRadMat facility at SPS, CERN. Online techniques such as Laser Doppler Vibrometry (LDV) and strain gauges are used to monitor the samples dynamic deformations and possible failure limits of simple and composite targets and for benchmarking thermo-mechanical calculations of beam-induced stress and strain.

## 3He polarization for JCNS instrumentation

ID 405

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An in-situ polarization analyzer has been in operation for the MARIA magnetic reflectometer for each reactor cycle for over 5 years. Here it provides a continual polarization and a wide angle of  $\pm 6^\circ$  for a cold neutron beam that is fully decoupled for the sample environment conditions and magnetic field. This device has been used as the basis for additional polarizer devices, using what has been learned to develop improved devices. A polarizer for thermal-energy beams, such as on the planned TOPAS spectrometer, has been completed and tested on POLI for 0.89 Å neutrons. Similarly a very compact device based on a 50% reduced scale of the MARIA polarizer is in development for KWS1. The magnetic cavity of this device, which is only 39cm long is completed and has been used for experiments with off-line polarized 3He cells in conjunction with a newly procured 3T, longitudinal or horizontal, sample magnet providing good magnetic performance in both field configurations. Finally a project is continuing to make a very high angular coverage,  $\pm 17^\circ$ , device based on a compensated solenoid geometry for KWS2. The status of the various projects and device performance will be presented.

## Spin Hall Magnetoresistance in a Canted Ferrimagnetic Insulator

ID 406

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The interplay of charge and spin currents at the interface between ferrimagnetic insulators and paramagnetic metals gives rise to novel spintronic effects, such as the recently discovered spin-Hall magnetoresistance (SMR). The effect was described as a result of interfacial spin mixing, i.e., of a spin-angular-momentum exchange between the magnetization in the ferrimagnet  $\text{Y}_3\text{Fe}_5\text{O}_{12}$  and the spin polarization of the conduction electrons in paramagnetic Pt [1]. We study this effect in ferrimagnet/normal metal bilayers, comparing the response in the collinear and canted magnetic phases of  $\text{Gd}_3\text{Fe}_5\text{O}_{12}$ . In the collinear magnetic phase, where the sublattice magnetic moments are all aligned along the same axis, we observe the conventional SMR. In the canted phase, however, the SMR changes sign [2]. Using element-selective X-ray absorption and X-ray magnetic circular dichroism experiments, we understand these observations in terms of the magnetic field and temperature dependent reorientation of magnetic moments on the different magnetic sublattices of  $\text{Gd}_3\text{Fe}_5\text{O}_{12}$  [2]. This enables a magnetotransport-based investigation of non-collinear magnetic textures. This work is supported by the European Synchrotron Radiation Facility (ESRF) via HE-3784, HC-1500, and HC-2058, as well as the Deutsche Forschungsgemeinschaft (DFG) via SPP 1538.

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## Structural and electronic correlation in hole-doped $\text{Pr}_{2-x}\text{Sr}_x\text{NiO}_{4+d}$

ID 412

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Co-doped  $\text{Pr}_{2-x}\text{Sr}_x\text{NiO}_{4+d}$  is one of the exotic family members belonging to the nicketes for many different reasons. Like h-Tc cuprates and cobaltates superconductors, this family of nicketes also shows very complicated incommensurability (epsilon) of charge and spin ordering which can be explained via dis-commensuration/mixing of stripe and checkerboard ordered phases. In addition to that, the high oxygen mobility, which has been explained in this compound via phonon assisted diffusion mechanism, give rise to intriguing complexity as the interstitial oxygen gets ordered on a very long range scale giving O-supercell volume of approximately ( $70.000 \text{ Angstrom}^3$ ). A series of compounds including pure sr-doped  $\text{Pr}_{1.5}\text{Sr}_{0.5}\text{NiO}_4$ , co-doped  $\text{Pr}_{1.875}\text{Sr}_{0.125}\text{NiO}_{4+d}$  and oxygen doped  $\text{Pr}_2\text{NiO}_{4.25}$  have been investigated by synchrotron and neutron scattering. The preliminary findings show in all cases different incommensurability though the nominal hole concentration ( $n_h=x+2d$ ) is expected to be the same. The oxygen containing samples show in-plane incommensurate modulation with vector  $\sim q(0.521, 0.805, 0)$  with satellites detected upto 4th order. We will discuss  $\text{Pr}_{2-x}\text{Sr}_x\text{NiO}_{4+d}$  a perfect model system to show the effect of oxygen ordering on the charge and spin correlations and their related excitations.

## New insights on the identification of iron structures in Fe-N-C catalysts by NIS and Mössbauer spectroscopy

ID 414

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In proton exchange fuel cells (PEFC) the hydrogen oxidation reaction and oxygen reduction reaction are catalysed by platinum-based catalysts. However, for economic reasons it is anticipated to replace platinum on the cathode for the oxygen reduction reaction (ORR). Fe N C catalysts are the most prominent substitute. Molecular FeN<sub>4</sub> centres were identified by different groups as ORR active sites [1-5]. However, the oxidation state and structure remains under debate, but it is crucial for further improvement of Fe-N-C catalysts. Recently, we were able to show that sulphite ions can be used as poison for Fe-N-C catalysts [6]. The comparison of different catalysts gives indications of systematic changes in the iron signature. However, due to the ambident character of sulphite ions the interpretation is not straight forward. In order to get additional insights, temperature dependent Mössbauer spectroscopy and Nuclear Inelastic Scattering (NIS), were used to refine our model of iron sites present in these catalysts. DFT calculations are used to modulate the partial density of iron states (pDOS). On the basis of this we are able to identify some identic and some changing iron sites in the asprepared and poisoned catalyst, that gives new insights on the nature of ORR activity in Fe-N-C catalysts. Acknowledgement: The results based on the beamtimes at PETRA III (I-20160285 and I-20170383). Financial support by the BMBF within the cooperation project NUKFER (05 K16 RD1), Fe-N-CStRedO (03XP0092) and from the TU Darmstadt Graduate School Energy Science and Engineering (ESE, GSC1070) is gratefully acknowledged. The authors are grateful for the beamtimes at the P01 beamline.

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**“Structure research” activities at MLZ**

ID 416

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Crystal structure defines properties and functionality for different/various classes of materials. In the task of the crystal and magnetic structure determination the neutron diffraction has been proven as a powerful research tool combining more than 100 years development of diffraction techniques (since Max von Laue experiments) along with unique (and well known) features of neutron scattering. A wealth of neutron diffraction instrumentation as a probe of long-range atomic and magnetic orders from single crystal and powder samples at a variety of environmental conditions is available at MLZ, namely high-resolution and engineering diffractometers SPODI and STRESS-SPEC; hot, thermal and cold (macromolecular) single crystal diffractometers HEIDI, RESI and BioDIFF; diffuse instrument DNS and a pool of triple axis spectrometers. A number of new instrumental developments (POWTEX, SAPHIR, ERWIN) are on their way to strengthen diffraction at MLZ even further. There is a broad spectrum of structure-related scientific activities at MLZ e.g. (i) electrochemical energy storage systems and related materials; (ii) modern ferroelectrics; (iii) multiferroic materials and interrelation of the ferroic degrees of freedom; (iv) new generation engineering and shape memory alloys; (v) biological macromolecules; (vi) rock-forming minerals and glasses. Current contribution aims to present a brief introduction and report on interests, activities and achievements of MLZ Group “Structure Research”.

## Multi-Modal, Multi-Dimensional, Correlative Imaging: News from the GINIX

ID 419

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The Göttingen Instrument for Nano-Imaging with X-rays is the nanofocus-setup at the coherence beamline P10 at PETRA III, DESY Hamburg. It features a 300 nm Kirkpatrick-Baez mirror system as a prefocus for X-ray waveguide (WG) optics; these WGs act as coherence filter and cleanup the X-ray beam from artefacts in the illumination. In holography mode, sub-50 nm resolution of biological / organic specimens becomes possible in three dimensions. In contrast, scanning nano-SAXS in the focused beam provides local access to physical quantities, e.g. orientations and sizes of collective scatterers such as sarcomeric structures. A super-resolving optical fluorescence microscope has been combined into the X-ray microscope. Using STimulated Emission Depletion (STED), the optical path of the setup allows for imaging of labelled molecules at a resolution scale of 100 nm; combined with the X-ray holography, the labelled functional components of biological cells (here: actin cytoskeleton in cardiac tissue cells) can be correlated to the electron density. Then, the nano-SAXS measurements contribute spatially resolved scattering information. We present a first correlative analysis combining STED and X-ray techniques on neonatal cardiac tissue cells. We can infer that the actin filaments, which are fluorescently labelled can be traced using STED, correlate to a significant extent with the filaments as segmented in the holographic X-ray image. From the nano-SAXS analysis, the filaments stand out by their anisotropic scattering, and the preferential orientation is quantified.



## NICOS - an instrument control framework

ID 422

**KRUEGER, Jens**

*BRANDL, Georg; FAULHABER, Enrico (TU München / Forschungs-Neutronenquelle Heinz Maier-Leibnitz, Germany); FELDER, Christian (MLZ); PEDERSEN, Bjoern (FRM II, TU München); RAINOW, Stefan (MLZ); FLEISCHHAUER-FUSS, Lydia (MLZ); STEFFENS, Alexander (MLZ); BIYANI, Nikhil (PSI)*

NICOS (Networked Instrument COntrol System) has all components needed to easily create an instrument control software. Due to the large number of configuration options it can be tailored to individual instruments' requirements. There is a script execution component, components to create a history of measured data as well as of nearly all parameters of the instrument. A highly configurable graphical user interface with a lot of different modules allows instrument specific design. To display the status of the instrument the most important instrument parameters may be displayed on a screen or via an internet browser. A number of standard commands exist, and instrument specific commands are possible. Various middleware systems currently TACO, TANGO and EPICS as well as protocols for plug-and-play integration of decoupled embedded systems, e.g. sample environments and further measurement options are supported. A newly developed protocol for cross facility integration of sample environment named SECoP has been integrated. Nearly all components are written in Python which gives a high degree of platform independence (Linux, Windows, macOS). It also allows easy implementation of new features (devices, commands, data sinks, and GUI components). Python is also used as the scripting language. At the MLZ, NICOS is now in use at around 20 instruments. An international collaboration with the SINQ at PSI and the ESS has started.

## High energy synchrotron radiation for the development of tungsten fibre-reinforced tungsten composites

ID 432

**RIESCH, Johann (Max-Planck-Institut für Plasmaphysik)**

*BUFFIERE, Jean-Yves (GEMPPM INSA Lyon 20 Av. A); COENEN, Jan (Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung – Plasmaphysik); ELGETI, Stefan (Max-Planck-Institut für Plasmaphysik); HÖSCHEN, Till (Max-Planck-Institut für Plasmaphysik); JASPER, Bruno (Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung – Plasmaphysik); LE BOURLLOT, Christophe (GEMPPM INSA Lyon); LINSMEIER, Christian (Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung – Plasmaphysik); LUDWIG, Wolfgang (GEMPPM INSA Lyon); DI MICHIEL, Marco (European Synchrotron Radiation Facility); SCHEEL, Mario (Synchrotron Soleil); YOU, Jeong-Ha (Max-Planck-Institut für Plasmaphysik); NEU, Rudolf (Max-Planck-Institut für Plasmaphysik)*

Due to its unique property combination, tungsten would be the ideal material for the highly loaded areas in a future fusion power plant. However, tungsten is brittle up to very high temperatures and prone to operational embrittlement. Tungsten fibre-reinforced tungsten composites utilize extrinsic toughening mechanisms similar to ceramic fibre-reinforced ceramics and therefore can overcome the brittleness problem of tungsten. The proof that this principle can solve the mentioned problems was conducted in the past years. In this contribution, we present the use of high energy synchrotron radiation in this process. The mechanisms leading to improved toughness have been characterized during mechanical tests in combination with high resolution high energy synchrotron tomography at the ESRF in Grenoble. Both tension and bending tests have been performed for the case of ductile (as-fabricated) as well as the brittle (heat treated) W fibres on model systems containing a single fibre surrounded by a tungsten matrix. A spatial resolution down to 3  $\mu\text{m}$  allowed a clear identification of active toughening mechanisms and their evolution during the test. The tomographic study in combination with a detailed comparison to analytical methods allowed a correlation of macroscopic material behaviour to microscopic mechanisms. Furthermore, we will present the first results of the analysis of residual stress present in the material after fabrication by synchrotron diffraction studies.

## Multiscale X-Ray Analysis of Biological Cells and Tissues by Scanning Diffraction and Coherent Imaging

ID 444

**NICOLAS, Jan-David** (Georg-August-Universität Göttingen)

*BERNHARDT, Marten* (Georg-August-Universität Göttingen); *SCHLICK, Susanne* (Universitätsmedizin Göttingen); *TIBURCY, Malte* (Universitätsmedizin Göttingen); *ZIMMERMANN, Wolfram-Hubertus* (Universitätsmedizin Göttingen); *KHAN, Amara* (Max-Planck- -Institut für Experimentelle Medizin); *MARKUS, Andrea* (Max-Planck-Institut für Experimentelle Medizin); *ALVES, Frauke* (Max-Planck-Institut für Experimentelle Medizin); *TOISCHER, Karl* (Universitätsmedizin Göttingen); *JOPPE, Karina* (Universitätsmedizin Göttingen); *LINGOR, Paul* (Universitätsmedizin Göttingen)

Recent improvements in the quality of focusing optics for X-rays has greatly advanced structural imaging of soft biological materials. We have applied scanning X-ray diffraction, a method that involves raster-scanning of a microfocused X-ray beam and collecting a series of scattering patterns, on cardiac cells and tissue to image variations in the nanostructure of cardiac muscle. It is shown how the spacing of the myosin filaments in muscle varies throughout the tissue, but also within single cardiac muscle cells and how external factors influence the filament spacing. In many of these experiments, cumbersome scanning of large tissue areas can be spared if holographic imaging is used in conjunction with a diffraction experiment. By means of holographic imaging, the electron density, encoded in the phase shift of the sample, can be directly imaged. This low-dose imaging modality therefore offers a complementary contrast and can yield high-resolution images down to approximately 50 nm. It is shown how holographic imaging in 2D and 3D can be combined with a scanning X-ray diffraction experiment to image identical structures on multiple length scales. In the future, it can be expected that, with more brilliant radiation now offered by 4th generation synchrotrons and X-ray free electron lasers, structural imaging by means of holography and scanning diffraction will become a popular tool to image heterogeneous biological samples such as the aforementioned cardiac cells or tissue.



## Surface induced order in ionic liquids

ID 447

**MEZGER, Markus (Johannes Gutenberg University Mainz, Institute of Physics)**

*MARS, Julian (Max Planck Institut for Polymer Research, Mainz); BIER, Markus*

In supported ionic liquid (IL) phase catalysis, the chemical reaction takes place in a sub-micrometer IL film, wetting a solid support material with high surface area. To understand diffusion of reactants and products across the IL/vapor interface, knowledge of the interfacial structure is crucial. We employ X-ray scattering techniques to investigate the near surface structure on the molecular length scale. Depending on the molecular architecture of the ILs containing long aliphatic side chains, we observe pinning of bulk correlations at the surface [1] and surface induced smectic order [2]. In the case of surface induced smectic order, lamellar structures extending up to 30 nm into the isotropic bulk are observed. In microheterogeneous ILs, solvates aggregates site-specifically within high affinity domains. Nonpolar molecules, for example, are preferentially situated in the aliphatic regions. At liquid/vapor interfaces, this can strongly affect the near surface structure. For different IL based multi component systems, we observe the formation of monomolecular adsorbate layers and solvate induced surface phase transitions.

[1] M. Mezger et al., Proc. Natl. Acad. Sci. USA 110, 3733 (2013).

[2] J. Mars et al., Phys. Chem. Chem. Phys. 19, 2665 (2017).



## Wednesday – September 19<sup>th</sup>, 2018

Each field is linked to the session's start page. Just click on the title!

09:00	<b>Plenary Talk</b> First experiments at XFEL <i>R. Feidenhans'l</i>			2001
09:45	<b>Keynote</b> Interaction of highly charged ions with surfaces and 2D materials <i>F. Aumayr</i>			2001
10:15	Coffee break			
10:45	<b>Keynote</b> X-Ray spectroscopy of multifunctional oxides <i>M. Müller</i>			2001
11:15	<b>PS 8</b> Functional materials 0001	<b>PS 9</b> Catalysis and others 2001	<b>MS 6</b> Next generation large scale facilities 0350	
12:15	Lunch break			
13:45	<b>PS 8</b> Functional materials 0001	<b>PS 1</b> Instrumentation and methods 2001	<b>MS 6</b> Next generation large scale facilities 0350	
14:30	<b>Plenary Talk</b> The MLZ User Committee <i>A. Rennie</i>			2001
14:40	<b>Awards</b> Best Poster Award and Wolfram Prandl-Prize			2001
15:30	Closing			2001
16:00 - 18:00	Guided tours of the FRM II			Meeting point: Conference office



## European XFEL – a new facility for fundamental science

Wednesday, 09:00  
MW 2001  
ID 451

*FEIDENHANS'L, Robert (European XFEL)*

European XFEL is the most recent large scale research infra structure in Europe. The facility was taken into user operation in September last year and more than 500 users have already participated in week long experiments. The facility is a hard X-ray free electron laser and provides a very powerful X-ray beam for research. The facility serves the European user community by providing the possibility for performing new classes of experiments to investigate the structure and dynamics of matter on the atomic length and time scales. The facility encompasses a 3.5 km long tunnel from DESY in Hamburg/Bahrenfeld to Schenefeld in Schleswig-Holstein where the experimental hall is placed. The tunnel encloses a 2 km long superconducting accelerator operated by DESY and undulator radiation sources that produces an extremely powerful and ultra-short X-ray beam. The beam is guided through the 900 m long photon transport system into the experimental hall. The first two experimental stations have been performing experiments succesfully for about one year. In total six instrumental stations will be operating in 2019. In the talk the basic principles of the European X-FEL will be discussed and results of some of the first experiments will be shown.



## Interaction of highly charged ions with surfaces and 2D materials

Wednesday, 09:45  
MW2001  
ID 398

*AUMAYR, Friedrich (TU Wien)*

The large amount of energy deposited by the neutralization of highly charged ions during their impact on solid surfaces leads to electronic excitations. Depending on the material's response to the ion impact, phenomena like sputtering and the formation of individual nanostructures at the surface can occur. To pinpoint the energy deposition and its time dependence experimentally we recently used freestanding two-dimensional materials like graphene as a target. By adjusting the ions' kinetic energy the interaction time with the 2D-solid can be controlled. Spectroscopic measurements of the ions after transmission not only give insight into the mechanisms of a rapid charge exchange and deexcitation sequence but show that highly charged ions are well suited to investigate properties of 2D materials under extreme conditions on a femtosecond time scale.



## X-Ray Spectroscopy of Multifunctional Oxides

Wednesday, 10:45  
MW2001  
ID 431

*MÜLLER, Martina (FZ Jülich/ TU Dortmund)*

Beyond their traditional role as dielectrics, oxides are fascinating quantum materials showing a multitude of behaviours – such as (multi-)ferroic, memristive, nonlinear optical and topological effects – which are promising for potential memory, logic and sensoric functions. Oxide materials may enable the efficient transformation between electronic and chemical states – and as such hold a key towards the development of sustainable energy materials and future nanoelectronic technologies. We currently experience the intertwining of the fields of quantum materials and semiconductor physics, and the emerging field of complex oxide heterostructures applies the prosperous experimental tools and concepts of semiconductor physics to the oxides –which exhibit even richer and tunable physical properties. The collective properties of the constituent electrons in complex oxide heterostructures give rise to exotic physical states, emerging phenomena and novel device functionalities, that have no counterpart in any other material systems. We will review the significance of X-Ray spectroscopy techniques for investigating electronic, chemical, magnetic states and phase transitions in functional oxides. To date, research activities using photon-based methodologies are striking for both attaining ground-, metastable- or transient states with the perspective to tune and control novel functionalities in quantum oxides.



## The User Committee of the Heinz Maier-Leibnitz Zentrum

Wednesday, 14:30  
MW2001  
ID 316

*RENNIE, Adrian (Uppsala University)*

The Heinz Maier-Leibnitz Zentrum in Garching is a major facility for science with neutron scattering and for related experiments. As an organisation that has many user experiments as a core part of the success, the input from the community is important. The management of the MLZ have recently established a User Committee to provide better communication: the committee has the mission to propose new strategic ideas and procedures to the MLZ Directorate with the ultimate goal to improve the MLZ user satisfaction. To fulfil this overall aim, the committee seeks to receive the broadest possible range of input from users. Any ideas that might affect the successful outcome of proposed research that concern handling of proposals, planning experiments, instruments, support laboratories, the control and analysis software or other related matters are welcome. Recognising that some specific issues might be best handled confidentially, contact can be made directly with any of the user committee members. All of the committee members welcome contact (see <https://mlz-garching.de/user-office/user-committee.html>) . The committee members and their particular areas of expertise will be presented briefly. The committee, elected by the users, is independent of the MLZ staff and management. It has regular meetings and brings matters raised to the attention of the MLZ Directorate.



**Wednesday – September 19<sup>th</sup>, 2018**  
**11:15-12:15**

***Parallel session 9***  
***Catalysis and Others***

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:15-11:30**

*Oliver Forstner*

LISEL@DREAMS – The future of Accelerator Mass Spectrometry

**11:30-11:45**

*Thomas Sheppard*

Revealing Catalyst Complexity in 3D with Synchrotron X-ray Nanotomography

**11:45-12:00**

*Raul Garcia-Diez*

Characterization of nanostructured iridium oxide-based electrocatalysts using advanced x-ray analytics

**12:00-12:15**

*Jan-Dierk Grunwaldt*

Catalysts and reactors under dynamic reaction conditions for energy storage and energy conversion

## LISEL@DREAMS – The future of Accelerator Mass Spectrometry

Wednesday, 11:15  
MW2001  
ID 354

**FORSTNER, Oliver (Friedrich-Schiller-Universität Jena)**

*VON BORANY, Johannes (Helmholtz -Zentrum Dresden-Rossendorf, Institute of Ion Beam Physics and Materials Research); MERCHEL, Silke (HZDR); RUGEL, Georg (Helmholtz-Zentrum Dresden-Rossendorf); WENDT, Klaus (Johannes Gutenberg-Universität Mainz)*

LISEL (Low energy Isobar SEparation by Lasers) is a future project at the DREAMS (DREsden Accelerator Mass Spectrometry) facility to widen the applications of AMS by extending the range of measurable (radio-) nuclides. AMS has proven to be a versatile tool capable of detecting a large number of long-lived radionuclides at the ultra-trace level – i.e. isotope ratios down to  $10E-16$ . However, being a mass spectrometric method, it is limited by the presence of strong isobaric background. To overcome this limitation, we propose to remove the isobars already at the low-energy side by laser photodetachment. This method allows to selectively neutralize isobars by laser radiation, leaving the ions of interest intact. First studies were performed at the University of Vienna and gave promising results [1,2] for the easier to be measured low-mass AMS isotopes Al-26 and Cl-36. Within the LISEL project this method will be for the first time applied to an AMS facility based on a 6 MV tandem accelerator. The first isotopes to be addressed with the new method will be Mn-53 and Fe-60. Both are currently only measurable at AMS facilities with more than 10 MV terminal voltage (currently available only at the ANU in Canberra or the LMU and TU Munich in Garching). Further on we foresee to apply this method to other rare isotopes, making LISEL@DREAMS a versatile machine for “all” isotopes. This will subsequently widen the applications and also the user community.

[1] Forstner, O. et al., Nucl. Instr. and Meth. B 361 (2015) 217.

[2] Martschini, M. et al., Int. J. Mass Spectrom., 415 (2017) 9.



## Revealing Catalyst Complexity in 3D with Synchrotron X-ray Nanotomography

Wednesday, 11:30  
MW2001  
ID 232

**SHEPPARD, Thomas (Karlsruhe Institute of Technology (KIT))**

*FAM, Yakub (Karlsruhe Institute of Technology); BECHER, Johannes (Karlsruhe Institute of Technology); DIAZ, Ana (Paul Scherrer Institut); HOLLER, Mirko (Paul Scherrer Institut); SCHROPP, Andreas (DESY); SCHROER, Christian (DESY / Uni. Hamburg); GRUNWALDT, Jan-Dierk (KIT)*

Catalysts are complex materials whose structure and reactive behaviour are closely interlinked. All length scales are relevant in catalysis, from metal nanoparticles, to porosity (micro- meso- and macropores), to global structure (e.g. grains, pellets or monoliths). While electron microscopy is well suited for analysis at (sub)nanometre resolution, this is often limited to small fields of view, thin sample lamella, and high vacuum conditions. Sample preparation is an important consideration, ideally delicate structural features should be preserved and measurements performed non-invasively. Developments in hard X-ray imaging using synchrotron radiation, particularly ptychographic X-ray computed tomography (PXCT), allow non-invasive measurement of large sample volumes with spatial resolutions unmatched by other X-ray microscopy methods. Here we demonstrate the derivation of structural information from two diverse catalyst systems using PXCT, (i) hierarchical nanoporous gold, (ii) Pt-alumina exhaust gas catalyst monoliths. In both cases micrometre-sized samples were measured, with 3D isotropic spatial resolutions of 13 and 35 nm, respectively. The 3D volumes obtained allowed direct and non-invasive quantification of catalyst surface area, pore size distribution and pore network topology. PXCT is highlighted as a method with uniquely high potential in catalysis research, which will further benefit greatly from the advent of fourth generation synchrotron light sources.

## Characterization of nanostructured iridium oxide-based electrocatalysts using advanced x-ray analytics

Wednesday, 11:45

MW2001

ID 238

**GARCIA-DIEZ, Raul (Renewable Energy, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH (HZB))**

*WILKS, Regan G. (Renewable Energy, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH (HZB)); BÄR, Marcus (Renewable Energy, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH (HZB)); SIEBEL, Armin (Technische Universität München, Chair of Technical Electrochemistry, Department of Chemistry and Catalysis Research Center); WEIß, Alexandra (Technische Universität München, Chair of Technical Electrochemistry, Department of Chemistry and Catalysis Research Center); BERNT, Maximilian (Technische Universität München, Chair of Technical Electrochemistry, Department of Chemistry and Catalysis Research Center); YANG, Wanli (Advanced Light Source (ALS), Lawrence Berkeley National Laboratory); GASTEIGER, Hubert A (TUM)*

Iridium oxide ( $\text{IrO}_2$ ) has been suggested as a promising candidate for efficient water oxidation, and thus catalyst materials based on nanostructured iridium (hydr)oxide have recently been developed and commercialized. In this work, we present a comprehensive spectroscopic study of commercially available iridium oxide-based electrocatalysts (“nano- $\text{IrO}_2$ ” and “ $\text{IrO}_2@ \text{TiO}_2$ ”) using advanced x-ray analytics at the synchrotron sources BESSY II and ALS. The spectroscopic study of these materials at the oxygen K-edge revealed a high-resolution fingerprint related to the reorganization of local electronic states presumably due to a varying degree of crystallinity, as suggested by the comparison with an extensively characterized rutile-like  $\text{IrO}_2$  and an amorphous iridium oxide. Furthermore, detailed information about the hybridization of electronic states in the valence band was obtained combining the atom specificity of soft x-ray absorption (XAS) and emission spectroscopy (XES) with the surface sensitivity of x-ray photoelectron spectroscopy (XPS). The study was completed comparing the spectroscopic results with diffraction and microscopy measurements, in which the heterogeneous nature of the nanostructured iridium oxide-based materials is confirmed, discerning contributions from a rutile-type structure and an amorphous phase. The latter phase has been implicated in the high catalytic activity of these materials in the sluggish oxygen evolution reaction.

## Catalysts and reactors under dynamic reaction conditions for energy storage and energy conversion

Wednesday, 12:00

MW2001

ID 380

*GRUNWALDT, Jan-Dierk (Karlsruher Institut für Technologie)*

*MUTZ, Benjamin (Karlsruhe Institute of Technology); KALZ, Kai (Karlsruhe Institute of Technology); LICHTENBERG, Henning (KIT); SERRER, Marc-Andre (KIT)*

Power-to-X technologies for conversion of electric power into chemical energy play a key role in many concepts for a future energy supply based on renewable sources. A good example is CO<sub>2</sub> hydrogenation to methane. Such reactions have extensively been studied under steady state conditions. However, for utilization of power from fluctuating renewable sources the reactors and especially the catalyst systems need to tolerate a dynamic supply of energy and reactants. The response of the catalyst to such dynamic changes is hardly known. [1] X-ray and neutron based techniques are ideal tools for monitoring structural dynamics of catalysts under changing reaction conditions, i.e. while the catalyst is working. Recently, we studied dynamic changes in the structure and performance of Ni-based catalysts during methanation of CO<sub>2</sub> while modulating the H<sub>2</sub> supply using operando X-ray absorption spectroscopy (XANES and EXAFS). We observed that an interruption of the H<sub>2</sub> feed led to some extent to oxidation of the Ni particles resulting in a deactivation of the catalyst.[2] Further studies using Quick-EXAFS showed that a higher modulation frequency had a less pronounced effect on the catalyst deactivation because oxidation/reduction effects occurred mainly on the surface of the Ni particles.[3]

[1] K. Kalz et al., Chem. Cat. Chem. 17 (2017) 9; SPP-Homepage: [www.spp2080.org](http://www.spp2080.org).

[2] B. Mutz et al., J. Catal. 327 (2015) 48.

[3] B. Mutz et al., Catalysts 7 (2017) 279.



**Wednesday – September 19<sup>th</sup>, 2018**  
**11:15-12:15**

## ***Parallel session 8***

# ***Functional materials and materials science***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:15-11:30**

*Klaus Habicht*

Phonon Lifetimes in the Perovskite Model-Thermoelectric SrTiO<sub>3</sub>

**11:30-11:45**

*Daria Noferini*

Neutron scattering for sustainable energy materials: investigations of proton dynamics in acceptor-doped barium zirconates

**11:45-12:00**

*Luca Silvi*

A quasielastic and inelastic neutron scattering study of the alkaline and alkaline-earth borohydrides LiBH<sub>4</sub>, Mg(BH<sub>4</sub>)<sub>2</sub> and the mixture LiBH<sub>4</sub>+Mg(BH<sub>4</sub>)<sub>2</sub>

**12:00-12:15**

*Philipp Marzak*

Neutron Depth Profiling and PGAA of Electrodeposited Prussian Blue Analog Thin Film Electrodes



## Phonon Lifetimes in the Perovskite Model-Thermoelectric SrTiO<sub>3</sub>

Wednesday, 11:15  
MW0001  
ID 319

**HABICHT, Klaus (Helmholtz-Zentrum Berlin für Materialien und Energie)**

*FRITSCH, Katharina (Helmholtz-Zentrum Berlin für Materialien und Energie); HOFMANN, Tommy (Helmholtz-Zentrum Berlin für Materialien und Energie); LU, Zhilun (Helmholtz-Zentrum Berlin für Materialien und Energie); KOJDA, Danny (Helmholtz-Zentrum Berlin für Materialien und Energie); RYLL, Britta (Helmholtz-Zentrum Berlin für Materialien und Energie); GROITL, Felix (Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institute); KELLER, Thomas (Max Planck Institute for Solid State Research)*

Computational materials design based on first-principle calculations holds a huge potential in identifying novel compounds with superior functional properties. To model thermal conductivity, the progress made with ab initio methods to date allows to calculate wavevector-dependent relaxation rates for every phonon mode in the Brillouin zone. Despite the tempting opportunities offered by computational methods it is mandatory to validate the underlying theoretical models which are often based on approximations to tackle the many-body problem. Experimental data on phonon lifetimes allows to test the predictive power of numerical calculations. We have used inelastic neutron scattering to measure phonon lifetimes in the perovskite model system SrTiO<sub>3</sub>. While triple-axis spectroscopy conducted at V2/FLEXX, BER II, HZB, convincingly demonstrates the anisotropy of phonon lifetimes, neutron resonance spin-echo spectroscopy performed at TRISP, FRM II, MLZ, allows to pin-point the phonon lifetimes on an absolute scale. Our experiments reveal a significant temperature dependence of lifetimes for transverse acoustic phonons. Brillouin zone boundary phonons show a pronounced increase in energy width below the antiferrodistortive phase transition at TC=105 K. Larmor diffraction measurements allow us to relate the reduced phonon lifetimes to additional phonon scattering at the domain walls.



## Neutron scattering for sustainable energy materials: investigations of proton dynamics in acceptor-doped barium zirconates

Wednesday, 11:30  
MW0001  
ID 146

*NOFERINI, Daria (Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz- Zentrum (MLZ))*

*KOZA, Michael Marek (Institut Laue-Langevin); KARLSSON, Maths (Chalmers University of Technology, Department of Chemistry and Chemical Engineering); EVENSON, Zachary; FOUQUET, Peter (Institut Laue-Langevin); FRICK, Bernhard (Institut Laue-Langevin); NILSEN, Gøran (ISIS Facility, STFC Rutherford Appleton Laboratory); RAHMAN, Seikh M. H. (Chalmers University of Technology, Department of Chemistry and Chemical Engineering)*

Proton conducting oxides are currently receiving considerable attention for their use as electrolytes in solid oxide fuel cells. One of the main challenges for these devices is to combine the advantages of a solid electrolyte with those of operational temperatures below 750°C, which is currently hampered by insufficient conductivities in the targeted T-range. The development of new electrolytes meeting the requirements for applications depends on a better understanding of the proton conduction mechanism. To this aim, systematic QENS investigations over a wide Q-E range were conducted on hydrated acceptor-doped barium zirconates, with focus onto the effect of the type and concentration of dopant atoms on the atomic-scale proton dynamics. The results show complex dynamics, arising from a distribution of different proton sites. At ps time scale, localized displacements interpretable as proton jumps and reorientations of the hydroxyl groups are disclosed. Faster motions are observed for the more distorted structures associated with higher doping levels. The analysis of the dynamics at longer time scales reveals long-range proton diffusion which can be described as a Chudley-Elliott jump-diffusion process. Higher doping concentrations lead to higher activation energies, but larger fractions of mobile protons. This new insight can be useful for the design of improved proton conductors for technological applications.



## A quasielastic and inelastic neutron scattering study of the alkaline and alkaline-earth borohydrides $\text{LiBH}_4$ , $\text{Mg}(\text{BH}_4)_2$ and the mixture $\text{LiBH}_4+\text{Mg}(\text{BH}_4)_2$

Wednesday, 11:45

MW0001

ID 75

**SILVI, Luca (Helmholtz-Zentrum Berlin)**

*ZHAO-KARGER, Zhirong (Karlsruhe Institute of Technology (KIT)); MAXIMILIAN, Fichtner (Karlsruhe Institute of Technology (KIT)); PETRY, Winfried (FRM II - TUM); LOHSTROH, Wiebke*

Quasielastic neutron scattering was used to investigate the low energy transfer dynamics of the complex borohydrides  $\text{Mg}(\text{BH}_4)_2$  in the  $\alpha$ - and  $\beta$ -modifications,  $\text{LiBH}_4$  in the low and high temperature crystal structure, and the 1:1 molar mixture of  $\text{LiBH}_4+\text{Mg}(\text{BH}_4)_2$ . All investigated compounds show a rich dynamic behavior below an energy range of  $\Delta E = 10$  meV with the superposition of rotational dynamics of the constituent  $[\text{BH}_4]^-$  anions and low lying lattice modes. For  $\text{Mg}(\text{BH}_4)_2$ , the rotational diffusion of the  $[\text{BH}_4]$  units was found to be much more activated in the metastable  $\beta$ -polymorph compared to the  $\alpha$ -phase, and the low lying lattice modes are even softer in the former crystal structure. In  $\text{Mg}(\text{BH}_4)_2$ , the structural phase transition is mainly governed by the lattice dynamics, while alkaline  $\text{LiBH}_4$  exhibits a transition of the  $[\text{BH}_4]$  rotations around the phase transition temperature. Ball milled  $\text{LiBH}_4+\text{Mg}(\text{BH}_4)_2$  remains a physical mixture of the parent compounds and each component retains its characteristic dynamic signature up to the melting temperature.

## Neutron Depth Profiling and PGAA of Electrodeposited Prussian Blue Analog Thin Film Electrodes

Wednesday, 12:00

MW0001

ID 293

**MARZAK, Philipp** (Technical University of Munich, Department of Physics)

*TRUNK, Markus (Technical University of Munich, Department of Physics); WERNER, Lukas (Technical University of Munich, Department of Physics); REVAY, Zsolt (Technical University of Munich, Heinz Maier-Leibnitz Zentrum); YUN, Jeongsik (Technical University of Munich, Department of Physics); GERNHAEUSER, Roman (Technical University of Munich, Department of Physics); GILLES, Ralph (Technical University of Munich, Heinz Maier-Leibnitz Zentrum); MÄRKISCH, Bastian (Technical University of Munich, Department of Physics); BANDARENKA, Aliaksandr (Technical University of Munich, Department of Physics)*

Electrodeposited Prussian Blue Analog (PBA) thin films serve as attractive electrode materials for aqueous Na-ion batteries as grid-scale energy storage systems (ESSs). Such ESSs based on secondary batteries are attractive to address the so-called generation versus consumption issue which is inevitably connected to the use of energy from naturally fluctuating renewable sources and the so-called Terawatt Challenge by 2050. Importantly, materials employed do not only need to provide sufficient energy and power density. They also need to be cost efficient, safe and environmentally benign [1,2] and need to be designed to last for at least ten years of operation in order to be economically viable. For achieving such long life times it is essential to understand the mechanisms of (dis)charging and degradation of the materials. Neutron depth profiling and prompt gamma activation analysis are used to determine the spatial distribution of alkali metal cations within, and the elemental composition of PBA thin films operated as intercalation compounds. Knowing how the cation distribution within the films and their chemical composition evolve with a change in state of charge will allow for better understanding the charge and mass transfer mechanisms within the electrodes so that optimized and more durable systems can be developed.

[1] Qian, J. et al. Adv. Energy Mater. 2018, 1702619.

[2] Wessells, C. et al. Nano Lett. 2011, 11, 5421-5425.



**Wednesday – September 19<sup>th</sup>, 2018**  
**11:15-12:15**

## ***Micro symposium 6***

### ***Next generation large scale facilities***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

**11:15-11:30**

*Alexander Föhlisch*

The Variable Pulse Length Storage Ring BESSY-VSR

**11:30-11:45**

*Stefan Schippers*

Atomic, Plasma Physics and Applications (APPA) at FAIR

**11:45-12:00**

*Thomas Brückel*

The HBS Project for a High Brilliance neutron Source

**12:00-12:15**

Discussions



## **The Variable Pulse Length Storage Ring BESSY-VSR**

**Wednesday, 11:15  
MW0350  
ID 427**

***FÖHLISCH, Alexander (HZB/U Potsdam); ON BEHALFT OF THE BESSY VSR PROJECT***

In the coming years BESSY II is undergoing its up-grade to BESSY-VSR, a novel approach to create in the Storage Ring long and short photon pulses simultaneously for all beam lines through a pair of superconducting bunch compression cavities. Pulse-picking schemes will allow each individual user to freely switch between high average flux for X-ray spectroscopy, microscopy and scattering and picosecond pulses up to 500 MHz repetition rate for dynamic studies. Thus BESSY-VSR preserves the present average brilliance of BESSY II and adds the new capability of user accessible picosecond pulses at high repetition rate. For the scientific challenges of quantum materials for energy, future information technologies and basic energy science BESSY-VSR is the multi-user Synchrotron Radiation facility that allows with the flexible switching between high repetition rate for picosecond dynamics and high average brightness to move classical 3rd generation Synchrotron Radiation science from the observation of static properties and their quantum mechanical description towards the function and the control of materials properties, technologically relevant switching processes and chemical dynamics and kinetics on the picosecond time scale. BESSY-VSR creates for the highly productive Synchrotron Radiation community a uniquely attractive multi user storage ring adding the soft X-ray picosecond dynamics up to MHz repetition rate at preserved average brilliance. In particular investigations on reversible dynamics and switching in molecular systems and materials are accessible in a non-destructive way. The investigations with X-rays from BESSY-VSR are highly complementary and compatible to dynamic studies conducted by users with optical lasers at their home universities and laboratories. BESSY-VSR represents also a missing link between the extreme average brilliance of ultimate storage rings and Free Electron Lasers.



**Atomic, Plasma Physics and Applications (APPA) at FAIR**

**Wednesday, 11:30  
MW0350  
ID 305**

***SCHIPPERS, Stefan (Justus-Liebig-Universität Gießen, I. Physikalisches Institut)***

The international Facility for Antiproton and Ion Research (FAIR, [www.fair-center.eu](http://www.fair-center.eu)) is currently under construction at the site of the GSI Helmholtzzentrum für Schwerionenforschung in Darmstadt, Germany. After its completion in 2024/2025 FAIR will provide heavy-ion beams of highest intensities to a diverse set of research fields such as hadron physics, nuclear astrophysics, atomic physics, plasma physics, materials science or radiation biology, to name a few. Various scientific collaborations have gathered under the four research pillars of FAIR, i.e., APPA, CBM, NUSTAR and PANDA. In my talk, I will focus on APPA (Atomic and Plasma Physics and Applications) which represents more than 600 scientists from more than 30 countries. Under the umbrella of APPA, the international collaborations SPARC (atomic physics), HED@FAIR (plasma physics) and BIOMAT (biophysics and materials science) pursue a broad interdisciplinary research program [Nucl. Instrum. Methods B 365 (2015) 680] which addresses all FAIR relevant aspects of the electromagnetic interaction and which employs nearly the entire research infrastructure of FAIR, in particular, the full ion-energy range - from rest up to several GeV - provided by the FAIR accelerators. Large parts of this research program are driven by BMBF-funded German university groups (see [appa-rd.fair-center.eu](http://appa-rd.fair-center.eu)) who bring in their expertise for developing, setting up and exploiting cutting-edge instrumentation at FAIR.



## The HBS Project for a High Brilliance neutron Source

Wednesday, 11:45  
MW0350  
ID 105

**BRÜCKEL, Thomas (Forschungszentrum Jülich GmbH)**

*GUTBERLET, Thomas (Forschungszentrum Jülich); RÜCKER, Ulrich; MAUERHOFER, Eric (Forschungszentrum Jülich GmbH); ZAKALEK, Paul (Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science (JCNS-2) and Peter Grünberg Institut (PGI-4), JARA-FIT); CRONERT, Tobias (Forschungszentrum Jülich GmbH); VOIGT, Jörg (Forschungszentrum Jülich); BAGGEMANN, Johannes (Forschungszentrum Jülich GmbH); DOEGE, Paul (Forschungszentrum Jülich GmbH)*

We will report on the progress of the HBS project for scalable compact accelerator driven high brilliance neutron sources, optimized for neutron scattering on small samples and realizable at reasonable costs. As more and more older research reactors are being permanently shut down, such sources can support a new network of small to medium size facilities for user recruitment, user training, method development, specialized experiments and sheer capacity, which is essential to underpin the flagship facilities of ILL and ESS. The sources are based on particle induced reactions and can range from university neutron laboratory scale to full fledged user facilities highly competitive to present day medium sized facilities. Starting from the science case, we will outline the basic setup of such HBS type sources, discuss the technical realization and present the expected instrument performances.



**Wednesday – September 19<sup>th</sup>, 2018**  
**13:45-14:30**

## ***Parallel session 1***

### ***Instrumentation and methods***

**MW2001**

*Each field is linked to the abstract's start page. Just click on the title!*

**13:45-14:00**

*Mathias Richter*

Application of multiple-orbit synchrotron radiation for electron time-of-flight spectroscopy

**14:00-14:15**

*Pieter Glatzel*

A new tender X-ray spectrometer at ESRF ID26

**14:15-14:30**

*Wojciech Roseker*

Nanosecond dynamics measured with Split-Pulse X-ray Photon Correlation Spectroscopy At Free Electron Laser Sources



## Metrology with Synchrotron Radiation

Wednesday, 13:45  
MW2001  
ID 332

**RICHTER, Mathias (Physikalisch-Technische Bundesanstalt)**

*ULM, Gerhard (Physikalisch-Technische Bundesanstalt)*

For more than 35 years, Germany's national metrology institute PTB uses synchrotron radiation for radiometry and further metrological tasks. Currently, the electron storage rings BESSY II and PTB's own Metrology Light Source (MLS) in Berlin-Adlershof are used for this purpose. Basic methods refer to source-based radiometry (operating the storage rings as primary source standards), detector-based radiometry (using cryogenic substitution radiometers as primary detector standards), and reflectometry. Apart from customer services, the work is performed within the framework of scientific cooperations with partners from industry and research. Topics are the characterization of space instruments and optics for EUV lithography, FEL photon diagnostics, nanometrology, and quantitative materials characterization by X-ray, VUV, and IR spectrometry. The contribution will give an overview of the facilities, capabilities, and activities in the field of metrology with synchrotron radiation at PTB.



## A new tender X-ray spectrometer at ESRF ID26

Wednesday, 14:00  
MW2001  
ID 292

**GLATZEL, Pieter (ESRF - The European Synchrotron)**

*ROVEZZI, Mauro (Univ. Grenoble Alpes, CNRS, IRD, Irstea, Météo France, OSUG, FAME, 38000 Grenoble, France); DETLEFS, Blanka (ESRF - The European Synchrotron); KOCSIS, Menhard (ESRF - The European Synchrotron); VAN VAERENBERGH, Pierre (ESRF - The European Synchrotron); MANCEAU, Alain (Univ. Grenoble Alpes, Univ. Savoie Mont Blanc, CNRS, IRD, ISTERre, 38000 Grenoble, France); VERBENI, Roverta (ESRF - The European Synchrotron)*

The energy range between 1.5 and 5 keV (tender X-rays) covers absorption edges of important elements whose electronic and magnetic structure is challenging to study in the hard or soft X-ray range. X-ray emission spectroscopy (XES) in combination with X-ray absorption spectroscopy (XAS) can provide sharper spectral features and allows studying the occupied and unoccupied density of electronic states thus providing a wealth of information. There is a rapidly growing interest in the X-ray spectroscopy community in XAS-XES. Tender X-ray emission spectrometers have been realized in different geometries. We recently commissioned at the ESRF an instrument in non-dispersive, scanning geometry that employs eleven Johansson crystals in combination with a gas proportional counter. The 80 mm long Si crystal wafers, cut along the (111) direction, are cylindrically bent to 0.5m radius in the meridional plane. The sagittal dimension is 25mm requiring a large detector surface of 50 x 25 mm<sup>2</sup>. The angular range of the instrument is 35 to 85 degrees. The presentation discusses the concept of the instrument and recent results.



## Nanosecond dynamics measured with Split-Pulse X-ray Photon Correlation Spectroscopy At Free Electron Laser Sources

Wednesday, 14:15  
MW2001  
ID 68

**ROSEKER, Wojciech (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany)**

*HRUSZKEWYCZ, Stephan (Argonne National Laboratory, Argonne, Illinois, USA); LEHMKÜHLER, Felix (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany); WALTHER, Michael (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany); SCHULTE-SCHREPPING, Horst (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany); LEE, Sooheyong (Korea Research Institute of Standards and Science, Daejeon, Rep. of Korea); OSAKA, Taito (RIKEN SPring-8 Center, Sayo-cho, Sayo-gun, Hyogo, Japan); STRÜDER, Lothar (PNSensor GmbH, München, Germany); Dr HARTMANN, Robert (PNSensor GmbH, München, Germany); SIKORSKI, Marcin (European X-Ray Free-Electron Laser Facility, Schenefeld, Germany); SONG, Sanghoon (LAC National Accelerator Laboratory, USA); ROBERT, Aymeric (LAC National Accelerator Laboratory, USA); FUOSS, Paul (LAC National Accelerator Laboratory, USA); Prof. SUTTON, Mark (McGill University, Montreal, Quebec, Canada); STEPHENSON, Brian (Argonne National Laboratory, Argonne, Illinois, USA); GRÜBEL, Gerhard (Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany)*

One of the important challenges in condensed matter science is to understand ultrafast, atomic-scale fluctuations that dictate dynamic processes in equilibrium and non-equilibrium materials. These fluctuations can be measured on the relevant time scale by time-correlating coherent scattering speckle patterns taken with ultrashort X-ray pulses from a free electron laser (FEL) in an X-ray Photon Correlation Spectroscopy (XPCS) experiment if the time separation of the X-ray pulses can be controlled on that very timescale. Here, we report an important step towards reaching that goal by using a prototype perfect crystal-based split-and-delay system, capable of splitting individual X-ray pulses and introducing femto- to nanosecond time delays. We show the results of the first ultrafast XPCS experiment[1] at LCLS where split X-ray pulses were used to measure the dynamics of gold nanoparticles suspended in a liquid and verify the validity of the Stokes-Einstein relation on ns-ps timescales. We show how reliable speckle contrast values can be extracted even from very low intensity FEL speckle patterns by applying maximum likelihood fitting, thus demonstrating the potential of a split-and-delay approach for dynamics measurements at FEL sources. This capability promises to elucidate the underlying dynamics of a wide variety of systems and will enable the discovery of new physical processes therein.

[1] Roseker, W. et al. Nature Communications 9,1704 (2018).



**Wednesday – September 19<sup>th</sup>, 2018**  
**13:45-14:30**

## ***Parallel session 8***

# ***Functional materials and materials sciences***

**MW0001**

*Each field is linked to the abstract's start page. Just click on the title!*

**13:45-14:00**

*Sandro Szabo*

Structure and Dynamics in Bulk Metallic Glass forming liquids with minor additions

**14:00-14:15**

*Debashis Mukherji*

How neutron and synchrotron measurements are aiding Co-Re alloy development for gas turbines and supplementing electron microscopy studies

**14:15-14:30**

*Johannes Costa*

Inelastic Neutron Scattering on FeNiCo High-Entropy Alloys



## Structure and Dynamics in Bulk Metallic Glass forming liquids with minor additions

Wednesday, 13:45  
MW0001  
ID 58

**SZABO, Sandro (Heinz Maier-Leibnitz Center (MLZ) and Physics Department, Technical University of Munich, Garching, Germany)**

*EVENSON, Zachary; YANG, Fan (Institute of Materials Physics in Space, German Aerospace Center (DLR), Köln, Germany); MEYER, Andreas (German Aerospace Center); PETRY, Winfried (FRM II - TUM)*

Enhancing the glass-forming ability of alloys via the minor addition of specific elements is a technique currently at the forefront of BMG design. The addition of only 4 at.% Al or Ti to binary Zr<sub>50</sub>Cu<sub>50</sub> increases its critical casting thickness from 2 mm to at least 5 mm. However, the physical picture of how minor additions improve the glass-forming ability of alloys is still largely incomplete. Using in-situ synchrotron X-ray diffraction and quasi-elastic neutron scattering (QENS), we investigate the interplay between melt structure and mass transport quantities like viscosity and self-diffusion, as well as the solidification behavior of the (Zr<sub>50</sub>Cu<sub>50</sub>)<sub>96</sub>(Ti,Al)<sub>4</sub> alloys. Combined with electrostatic levitation (ESL), we are able to measure the liquid properties of these chemically reactive alloys over a large temperature range. At the liquidus temperature of Zr<sub>50</sub>Cu<sub>50</sub>, the selfdiffusivity in the investigated (Zr<sub>50</sub>Cu<sub>50</sub>)<sub>96</sub>Al<sub>4</sub> and (Zr<sub>50</sub>Cu<sub>50</sub>)<sub>96</sub>Ti<sub>4</sub> alloys is almost a factor of two lower than in Zr<sub>50</sub>Cu<sub>50</sub>. For the (Zr<sub>50</sub>Cu<sub>50</sub>)<sub>96</sub>Al<sub>4</sub> alloy, this is in line with the observation that minor Al addition leads to higher melt viscosities. However, despite the different microscopic dynamics, the average packing fractions of Zr<sub>50</sub>Cu<sub>50</sub> and (Zr<sub>50</sub>Cu<sub>50</sub>)<sub>96</sub>(Ti,Al)<sub>4</sub> melts are very similar. Hence, contributions from chemical short range order are expected to play an important role in the melt. The ternary alloys also exhibit different crystallization behavior compared to that of Zr<sub>50</sub>Cu<sub>50</sub>, which vary with overheating temperature and cooling rate. Both the sluggish dynamics and the complex solidification could contribute considerably to the improved glass-forming ability upon minor addition.

## How neutron and synchrotron measurements are aiding Co-Re alloy development for gas turbines and supplementing electron microscopy studies

Wednesday, 14:00

MW0001

ID 60

**MUKHERJI, Debashis (TU Braunschweig)**

*GILLES, Ralph (FRM2 TU Munich); STRUNZ, Pavel (Nuclear Physics Institute); BERAN, Přemysl (Nuclear Physics Institute); HOFMANN, Michael (FRM2 TU Munich); HOELZEL, Markus (FRM2 TU Munich); RÖSLER, Joachim (TU Braunschweig)*

Co-Re based alloys are being developed at the Technische Universität Braunschweig [1], to complement Ni-superalloys in gas turbine engines. Their main advantages are high melting temperature, very high specific strength, good ductility and good oxidation resistance. The Co-Re alloys are strengthened by a fine dispersion of carbides, particularly MC type TaC but unlike gamma-gamma prime based Ni-superalloys, they have a rather complex microstructure with many different phases present in diverse morphology and in different length scale. Additionally, the matrix Co phase undergoes an allotropic phase transformation at high temperatures (as high as 1200°C depending on alloy composition) from the low temperature hexagonal closed pack (hcp) Co structure to the high temperature face centered cubic (fcc) Co structure. In order to understand and study the complex phase relations and their effect on the mechanical properties, in the Co-Re alloy development we extensively used in-situ neutron and synchrotron measurements along with electron microscopy for the alloy development. Through some examples in this presentation, we will show how neutron and synchrotron measurements are helping in understanding how microstructures are developed in various Co-Re alloy composition and how they evolve on exposure to high temperatures. Such information was clearly not possible with microscopic analysis of the ex-situ heat treated Co-Re alloy specimens alone and thus neutron and synchrotron probes are providing a valuable tool for the alloy development. These measurements also allow the application of loading during the in-situ measurements at the high temperatures [2] and thereby allow to simulate conditions of loading during the service in the gas turbines.

[1] J. Rösler, D. Mukherji, T. Baranski (2007), Adv. Eng. Mater 9, 876-881.

[2] L. Kargea, R. Gilles, D. Mukherji, A. Stark, P. Beran, N. Schell, M. Hofmann, P. Strunz, J. Häusler, J. Rösler, Mat. Sci. .Eng., A 719 (2018) 124–131.



## Inelastic Neutron Scattering on FeNiCo High-Entropy Alloys

Wednesday, 14:15  
MW0001  
ID 167

*COSTA, Johannes (Physik-Department, Technische Universität München)*

*PETRY, Winfried (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München (TUM)); LEITNER, Michael (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München (TUM))*

High-entropy alloys, equiatomic alloys of five or more principal elements, have recently been the focus of much attention due to their superior structural properties. The stability of these materials is a matter of great concern for application, where it is still not settled whether the prototypical examples are thermodynamically stable phases or just metastable on accessible timescales. In this respect, a profound knowledge of lattice vibrations (phonons) is necessary as they are responsible for phase stabilities and thermodynamic properties, but also for other properties such as thermal conductivity. However, these issues have so far scarcely been studied directly. Equiatomic FeNiCo is of specific interest as a medium-entropy analogue of the prototypical Cantor alloy CoCrFeMnNi. In this class of materials, clearly also the role of magnetic effects has to be considered, with the richness of phenomena expected due to the interactions between the different atomic moments arranged on the face-centred cubic lattice with potential short-range order, and the interplay between magnonic and phononic excitations at elevated temperatures. Here we present the results obtained by inelastic neutron scattering, displaying the evolution of the vibrational spectra with temperature. We also comment on the possibility to observe short-range order via the elastically diffracted intensity. The experiments were performed at the time-of-flight spectrometer (TOFTOF) at the MLZ Garching.



**Wednesday – September 19<sup>th</sup>, 2018**

**13:45-14:30**

## ***Micro symposium 6***

### ***Next generation large scale facilities***

**MW0350**

*Each field is linked to the abstract's start page. Just click on the title!*

**13:45-14:00**

*Wilfried Wurth*

FLASH2020+ - a unique XUV and soft x-ray free-electron laser

**14:00-14:15**

*Andreas Schreyer*

News from the European Spallation Source

**14:15-14:30**

*Christian G. Schroer*

PETRA IV: The ultra-low emittance source project at DESY



**FLASH2020+ - a unique XUV and soft x-ray free-electron laser**

**Wednesday, 13:45  
MW0350  
ID 421**

***WURTH, Wilfried (DESY/Universität Hamburg)***

The DESY2030 strategy includes an ambitious development program for FLASH – FLASH2020+. FLASH is based on its superconducting accelerator currently the only high-repetition rate XUV and soft x-ray free-electron laser (FEL) facility worldwide. The goal of FLASH2020+ is to stay at the forefront of FEL technology in order to enable excellent science and ensure optimum conditions for users also in the future. The present FLASH2020+ design foresees to operate a multi FEL facility with two FEL lines. The first FEL line operated in self-amplified spontaneous emission (SASE) mode shall feature novel lasing schemes based on an advanced flexible undulator concept. The second line will be externally seeded while maintaining the high repetition rate of FLASH. Furthermore we plan to extend the wavelength range in the fundamental towards the O-K-edge and to provide variable polarization in fundamental and/or harmonics. Finally FLASH2020+ will extend the capabilities for time-resolved experiments using pump-probe schemes by generating ultra-short, sub-femtosecond pulses and by providing more flexible multi-colour pump schemes.



### News from the European Spallation Source

Wednesday, 14:00  
MW0350  
ID 426

#### *SCHREYER, Andreas (ESS)*

The European Spallation Source (ESS), which is currently under construction in Lund, Sweden, is designed to push the limits of research with neutrons to new horizons. ESS will open up new scientific opportunities which are complementary to those at X-ray sources. These will include unprecedented in-situ and in-operando experiments which are only possible with neutrons due to their special properties. After a short summary of the design and the specifications of the European Spallation Source 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 in 2023/24 and ramp up to 15 instruments by 2026.



**PETRA IV: The ultra-low emittance source project at DESY**

**Wednesday, 14:15  
MW0350  
ID 274**

***SCHROER, Christian G. (DESY / Uni. Hamburg)***

*AGAPOV, Ilya (DESY); BREFELD, Werner (DESY); BRINKMANN, Reinhard (DESY); Dr CHAE, Yong-Chul (DESY); CHAO, Hung-Chun (DESY); ERIKSSON, Mikael (MAX IV Laboratory); KEIL, Joachim (DESY); GAVALDA, Xavier Nuel (DESY); RÖHLSBERGER, Ralf (DESY / University of Hamburg); SEECK, Oliver; SPRUNG, Michael (DESY); TISCHER, Markus (DESY); WANZENBERG, Rainer (DESY); WECKERT, Edgar (DESY)*

The PETRA IV project aims at upgrading the present synchrotron radiation source PETRA III at DESY into an ultra-low emittance source. Being diffraction limited up to X-rays of about 10 keV, PETRA IV will be ideal for 3D X-ray microscopy of biological, chemical, and physical processes under realistic conditions at length scales from atomic dimensions to millimetres and time scales down to the sub-nanosecond regime. In this way, it will enable groundbreaking studies in many fields of science and industry, such as health, energy, earth & environment, transport, and information technology. The science case is reviewed and the current state of the conceptual design is summarised.