11th European NESY Winter-School & Symposium on Neutrons & Synchrotron Radiation

including topical highlight lectures on

Materials

Altaussee (Austria), 4-8 March, 2019

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The NESY Winterschool & Symposium is the bi-annual forum for Austrian neutron and synchrotron radiation (NESY) users with the aim to discuss new scientific achievements and to educate young academics. This traditional event was established in 1999 with the 1st *NESY Winterschool Planneralm*, which means that this year we organize already the **11th NESY Winterschool & Symposium** *and* can thus celebrate its **20-year anniversary**.

In the first couple of years this traditional event was a pure "*School*", where in tutorial lectures the large variety of NESY techniques available at Europe's large scale research facilities were introduced. In the last years, we kept the sequence of tutorials at the beginning and in the morning sessions, but the *School* part was more and more accompanied by scientific talks in the afternoon/evening forming a scientific "*Symposium"*. The underlying motivation is to give Austrian researchers a platform to discuss how the presented techniques are used within different research fields to solve important and timely scientific questions.

The morning sessions are largely reserved for **tutorials**, while the scientific sessions are held mainly in the late afternoon –after the skiing break- with **invited talks** and short **contributed scientific lectures**, and four evening sessions are reserved for **topical highlight lectures**. Finally, an informal, open-end **poster session** with drinks and discussions will conclude three meeting days. All in all, we have this year 49 contributions - 12 Tutorials, 4 invited keynote lectures, 6 invited talks, 8 contributed short lectures, 4 talks on research infrastructure and 15 posters - which means that the dominant part of the roughly 60 participants contribute actively to the success of the event.

The thematic focus this year is on **materials**, a very broad topic of ever increasing importance that covers the whole range from basic research to real industrial applications. We are very happy to have attracted excellent *international* keynote- and invited speakers, e.g., from the European neutron spallation source ESS, the synchrotron radiation sources ESRF and PETRA III, the neutron source FRM II, as well as from the Technion Haifa in Israel.

Several representatives from the strong Austrian materials research community presents their work ranging from very soft (biological) materials to very hard metals. But also from various other (non)-European countries young scientist present their research work. Hence, a broad range of scientific disciplines from, e.g., biology, physics, chemistry and materials science will be covered as well.

For the second time, the **2nd CERIC satellite event** will take place presenting activities of the European CERIC-ERIC initiative, where characterisation techniques complementary to the NESY techniques can be used on open access basis.

The program summarised in the following pages illustrates that after 20 years the **NESY Winterschool & Symposium** still follows the original idea -educating students and bringing the Austrian NESY community together-, but offers now also a platform for European young scientists to present their results gained on Europe's large scale *neutron*-, *synchrotron radiation* and *free electron laser* research facilities.

We are grateful to the BMBWF, specifically to MR Dr. Daniel Weselka for the continuous support of this event. Financial support from the facilities DESY/Petra III and ILL is gratefully acknowledged, as well as sponsoring from the companies, Anton Paar, and Bruker AXS. The donations from industry will not only allow us to bestow two young scientist awards (one for the best lecture and one for the best poster), but also to have some additional money for the traditional free drinks at the poster sessions. We also thank all tutorial speakers and invited speakers for agreeing to come on their own costs, and to many others who have contributed in one or another way. This helps us to keep the school fee for students and young scientists very moderate. We are most grateful to Heide Kirchberger for taking over a considerable part of the organisational issues. We are looking forward to an inspiring and enjoyable NESY Winterschool and Symposium 2019 in Altaussee.

Leoben, February 2019 Rainer T. Lechner & Oskar Paris

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Day	Time	Title	Speaker	Affiliation	p.
Mo, 4th March					
Morning		**CERIC Satellite Event**			
	11:00-12:00	Introduction to CERIC and the ACCELERATE project: Open acces and proposal writing	M. Girod	CERIC-ERIC, Trieste (IT)	
	12:00-13:00	*Lunch Break*			
	13:00-14:00	Seeing with Electrons: Introduction to Electron Microscopy	C. Ghica	National Institute of Materials Physics, Magurele (RO)	
	14:00-15:00	Ion Beam Analysis: From routine techniques to state of the art applications	M. Jaksic	Ruđer Bošković Institute, Zagreb (HR)	
	15:00-16:00	NMR Spectroscopy: Research of Materials and Biologically Important Molecules	P. Sket	Slovenian National Institute of Chemistry, SloNMR* Centre, Ljubljana (SLO)	

Day	Time	Title	Speaker	Affiliation	p.
Mo, 4th March		**Opening: NESY Winterschool & Symposium**			
Afternoon Session		**Sources & Instrumentations**	**Chair:** R.T. Lechner		
	16:00-16:30	Welcome Coffee & Tea			
	16:30-16:45	Conference opening	R.T. Lechner	Institute of Physics, MU Leoben (AT)	
Tutorial	16:45-17:45	Synchrotron Radiation – Instrumentation and Techniques	O. Paris	Institute of Physics, MU Leoben (AT)	1
	17:45-18:00	*Break*			
Tutorial	18:00-19:00	Neutrons - Instrumentation and Techniques	K. Hradil	X-Ray Center, TU Wien (AT)	2
		Dinner Break			
Evening Session		**Keynote Lecture**	**Chair:** O. Paris		
	20:00-21:00	The European Spallation Source: New Opportunities for Science	A. Schreyer	ESS, Lund (SE)	3
	21:00	**Posters & Drinks**			

Day	Time	Title	Speaker	Affiliation	p.
Tue, 5th March					
Morning Tutorials		**Diffraction & Scattering**	**Chair:** R. Resel		

	8:40-9:40	Scanning X-ray and Neutron Diffraction on Polycrystalline Materials	J. Keckes	Material Physics, ESI & MU Leoben (AT)	4
	9:40-9:50	*Break*			
	9:50-10:50	Why Neutrons? A Compilation of Special Occasions	E. Jericha	Atominstitut, TU Wien (AT)	5
	10:50-11:00	*Break*			
	11:00-12:00	SAXS/WAXD with focus on soft matter and in-situ SAXS	H. Peterlik	Faculty of Physics, Univ. Wien (AT)	6
		Lunch			
		Afternoon Break			
Afternoon Session		**Symposium**			
	16:40-17:10	Coffee & Tea	**Chair:** J. Keckes		
invited talk	17:10-17:40	Development and Characterisation of Advanced Intermetallic Titanium Aluminide Alloys by means of Diffraction and Scattering Techniques	P. Erdely	Dep. of Materials Science, MU Leoben (AT)	7
	17:40-17:55	20µm Cross-Sectional High-Energy Synchrotron X-Ray-Microdiffraction on Nitrided, Carburized and Shot-Peened Steels Reveals Near-Surface	S. Bodner	Chair of Materials Physics, MU Leoben (AT)	9

		Microstructure, Phase and Stress Gradients			
	17:55-18:10	Stress and Microstructure Distributions across Scratch Track Cross-Sections in a CrN-Cr bilayer on Steel Revealed by 50nm XRD	M. Meindlhumer	CD-Laboratory MU Leoben (AT)	11
	18:10-18:15	*Break*			
	18:15-18:30	Hexagonal Si-Ge, characterization of a new crystalline phase realized in nanowire structures	D. Ziss	Institute of Semiconductor and Solid State Physic, JKU Linz (AT)	13
invited talk	18:30-19:00	Synchrotron radiation is a powerful tool to study supercapacitor charging behaviour	C. Koczwara	Institute of Physics, MU Leoben (AT)	15
		Dinner Break			
Evening Session		**Keynote Lecture**	**Chair:** K. Hradil		
	20:00-21:00	Smart neutrons for in-situ und operando research in the field of batteries and high-temperature alloys	R. Gilles	FRM II, Munich (DE)	16
	21:00	**Posters & Drinks**			

Day	**Time**	**Title**	**Speaker**	**Affiliation**	**p.**
We, 6th March					
Morning Session		**Grazing Incidence Techniques & X-ray Spectroscopy**	**Chair:** H. Peterlik		
Tutorial	8:40-9:40	Grazing Incidence X-ray Scattering Techniques: Crystallisation of Molecules at Solid Surfaces	R. Resel	Institute of Solid State Physics, TU Graz (AT)	17
	9:40-9:50	*Break*			
invited talk	9:50-10:20	Diindenoperylene molecules on 2D MoS2 substrates with GIWAXS	N. Mrkyvkova	Institute of Physics, Slovak Academy of Science, Bratislava (SK)	19
	10:20-10:35	The Conundrum of Organic Dye-Silica Hybrids: How Can Neutrons and Synchrotron Radiation Techniques Assist Us?	H.M. Moura	Institute of Materials Chemistry, TU Wien (AT)	20
	10:35-10:45	*Break*			
Tutorial	10:45-11:45	X-ray Spectroscopy - a tool for material testing	P. Kregsamer	Atominstitut, TU Wien (AT)	22
		Lunch			
		Afternoon Break			

Afternoon Session		**Symposium**			
	16:40-17:10	Coffee & Tea	**Chair:** R. Resel		
invited talk	17:10-17:40	Studying Drugs with Synchrotron Radiation	O. Werzer	Pharma-cutical Sciences, Dept. Pharm. Technolog, KFU Graz (AT)	23
	17:40-18:55	In Situ GISAXS Investigation of PtNi Bimetallic Alloy under Operational Conditions of Fuel Cells.	M. Bogar	CERIC-ERIC / TU Graz, Trieste (IT)	24
	17:40-18:55	Bio-Inspired Band Gap Engineering of Hybrid Perovskite MAPbBr3	A. Lang	Technion, Haifa (IL)	25
	18:10-18:15	*Break*			
	18:15-18:30	Studying Beam-induced Dynamics in Amorphous Ionic Conductors	K. Holzweber	*Faculty of Physics, Dynamics of Condensed Systems, Univ. Wien (AT)*	*26*
invited talk	18:30-19:00	Contrast variation in SAXS for Material Science Application	S. Haas	*FS-PEX / DESY, Hamburg (DE)*	*27*
		Dinner Break			
Evening Session		**Keynote Lecture**	**Chair:** J. Keckes		

	20:00-21:00	Micro-/Nano-beam X-ray Diffraction & Scattering	M. Burghammer	*ESRF, Grenoble (FR)*	*28*

Day	Time	Title	Speaker	Affiliation	p.
Thu, 7th March					
Morning Session		**Extreme Conditions & Tomography**	**Chair:** H. Amenitsch		
Tutorial	8:40-9:40	Extreme condition research using synchrotron radiation: Extended stability of carbon dioxide above 1 Mbar	R. Miletich	Institut für Mineralogie und Kristallographie, Univ. Wien (AT)	29
	9:40-9:50	*Break*			
Tutorial	9:50-10:50	Introduction to X-ray Tomography	J.W.C. Dunlop	Chemistry and Physics of Materials, Univ. Salzburg AT	30
	10:50-11:00	*Break*			
invited talk	11:00-11:30	X-ray Tomography on Biological Materials: Temperature- and Water Triggered Seed Release of Banksias	M. Eder	MPI for Colloids and Interfaces, Potsdam (DE)	31
	11:30-11:45	Determining the microstructural characteristics in paper based materials from X-ray Tomography	E. Machado-Charry	Institute of Solid State Physic, TU Graz (AT)	32

		Lunch			
Afternoon Session		**Afternoon Break** **Austrian Large Scale Research Activities**			
	16:30-17:00	Coffe & Tea	**Chair:** E. Jericha	Atominstitut, TU Wien (AT)	
	17:00-17:15	Österreichs Beteiligungen an internationaler Großforschung	H. Rauch	ÖAW (AT)	
	17:15-17:30	ENSA - European Neutron Scattering Association	H. Abele	Atominstitut, TU Wien (AT)	
	17:30-17:45	ESUO - European Synchrotron User Organisation and LEAPS	R.T. Lechner	Institute of Physics, MU Leoben (AT)	
	17:45-18:00	Kompakt-synchrotron	K. Hradil	X-Ray Center, TU Wien (AT)	
	18:00-19:15	NESY Meeting			
		Dinner Break			
Evening Session			**Chair:** O. Paris	Institute of Physics, MU Leoben (AT)	
	20:00-21:00	Utilizing Synchrotron Radiation to Reveal the Structure-function Relation in Biogenic and Bioinspired Materials	B. Pokroy	Technion Haifa (IL)	34
	21:00	**Posters & Drinks**			

Day	Time	Title	Speaker	Affiliation	p.
Fr, 8th March					
Morning Tutorials		**Novel Techniques with Synchrotron Radiation & Neutrons**	**Chair:** J.W. C. Dunlop		
	8:40-9:40	In-situ / Pump-probe Experiments at the Austrian SAXS beamline	H. Amenitsch	Institute of Inorganic Chemistry,TU Graz @ ELETTRA, Triest (AT)	35
	9:40-9:50	*Break*			
	9:50-10:50	X-ray magnetic circular dichroism: basic principles and recent developments in time-resolved imaging	A. Ney	Solid State Physics Division, JKU Linz (AT)	
	10:50-11:00	*Break*			
	11:00-12:00	Physics with Neutrons	H. Abele	Atominstitut, TU Wien (AT)	
	12:00-12:30	Prizes & Closing	R.T. Lechner & O. Paris	Institute of Physics, MU Leoben (AT)	

Poster Contributions

	Title	Presenter	Affiliation	p.
P1	Deformation and Crystallization Kinetics of a Soft Magnetic Iron Based Bulk Metallic Glass	M. Antoni	Chair of Material Physics, MU Leoben (AT)	38
P2	Surface Crystallization Studies of 2-decyl-7-phenyl-[1]benzothieno[3,2-b][1]benzothiophene (Ph-BTBT-10)	W. Bodlos	Institute of Solid State Physics, TU Graz (AT)	39
P3	Synthesis and Characterization of Crystalline Shape-Anisometric Polymer Particles as Model Systems for Self-Assembled Materials	T. Cavalli	Materials Chemistry, TU Wien (AT)	40
P4	Zernike Phase Contrast Nanotomography of Low Absorbing Biological Functional Materials	S. Flenner	Helmholtz-Zentrum Geesthacht (DE)	42

P5	Measuring Ionic Conductivity in Rubidium Borate Glasses via IS and aXPCS	T. Fritz	Faculty of Physics, Univ. Wien (AT)	43
P6	Determination of Chromium and Zinc Speciation in Airborne Particulate Matter by X-Ray Absorption Near-Edge Structure	G. Gunchin	Atominstitut, TU Wien (AT)	44
P7	Phase Transition Behavior of an Asymmetric Benzothienobenzothiophene (BTBT) Derivative	S. Hofer	Institute of Solid State Physics, TU Graz (AT)	45
P8	In vitro degradation experiments over one month period on magnesium-based alloys for biodegradable implant application	D. Krüger	Institut für metallische Biomaterialien, HZ-Geesthacht, Hamburg (DE)	46

P9	A New Ferromagnetic Insulator with Low Damping: Growth, Magnetic and Structural Properties of Zn/Al Doped Nickel Ferrite	J. Lumetzberger	Institute of Semiconductor and Solid State Physics, JKU Linz (AT)	48
P10	Structural Differences of Human and Yak Hair	A. Müllner	Faculty of Physics, Univ. Wien (AT)	49
P11	Residual stress fields of single steel ball impacts in Al alloys	M.-A. Nielsen	Institut für Werkstoffforschung, HZ-Geesthacht, Hamburg (DE)	
P12	In situ experiment for selective laser melting	J. Rosigkeit	Helmholtz-Zentrum Geesthacht (DE)	
P13	The Software Package GIDVis and its Application to Rotating Grazing Incidence X-Ray Diffraction Data	B. Schrode	Institute of Solid State Physics, TU Graz (AT)	52

P14	Crystal Structure Solution from Thin Films: Indexation of Grazing Incidence X-ray Diffraction Patterns	J. Simbrunner	Department of Neuroradiology, MedUni Graz (AT)	53
P15	3D X-ray Diffraction Microscopy (3DXRD) Using High Resolution X-ray Nanodiffraction	H. Stieglitz	Helmholtz-Zentrum Geesthacht (DE)	

Synchrotron Radiation – Instrumentation and Techniques

Oskar Paris
Institute of Physics, Montanuniversität Leoben, Austria
oskar.paris@unileoben.ac.at

This tutorial aims at a very basic introduction into the field of Synchrotron Radiation, addressing in particular students and postdocs who are newcomers in the field and who are also not necessarily physicists. In other words, it will be kept simple!

The tutorial will start with a very brief introduction into the basic interaction of X-rays with matter. Then I will discuss the production of synchrotron radiation in the different "generations" of synchrotron sources. The most important parts of a typical synchrotron beamline will be discussed as well. Finally I give a short overview about the basic principles of some selected experimental techniques related to the actual topic of the school, namely "materials".

Neutrons – Instrumentation and Techniques

Klaudia Hradil
X-ray center, TU Wien, Austria
klaudia.hradil@tuwien.ac.at

The distinctive physical properties of neutrons make them a superior probe to investigate materials. As free particles without charge they can interact with the nucleus of the material and dependent on energy penetrate materials in the range of a couple of millimetres or even centimetres. As waves within wavelengths in the range of interatomic distances and energies in the range of atomic movements they are suited to reveal the atomic arrangement and lattice dynamics within crystal lattices. Due to their magnetic moment they also can probe the magnetic structure and spin dynamics. Hence, the experiments involved to investigate materials using neutrons are extensive. In a typical neutron experiment one observes the change of the direction or energy of the neutron beam by taking its way through the sample. Analysing this information provides the researcher with information about the structure, dynamics and composition of the materials under investigation.

Neutron techniques yield essential information in a large range of scientific fields. Some examples are analysing stress/strain fields for economic life-time assessments of materials in industry, analysis of the atomic arrangements within energy storage materials in operando conditions which yield a better understanding of these processes and therefore to more effective devices.

To get these results instrumentation techniques optimised to the respective scientific question are required. Aim of the lecture here is to give a review of techniques in neutron investigation by citing different examples in materials, health, environmental science and conservation science.

The European Spallation Source: New Opportunities for Science

Andreas Schreyer
European Spallation Source, P.O Box 176, SE-221 00 Lund, Sweden
andreas.schreyer@esss.se

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 two years later. Selected examples of new scientific opportunities in the field of materials and life science will be discussed.

The Photo shows a recent view of the ESS construction site

Scanning X-ray and Neutron Diffraction on Polycrystalline Materials

Jozef Keckes

Department of Materials Physics, Montanuniversität Leoben, 8700 Leoben, Austria

keckes@unileoben.ac.at

Polycrystalline materials exhibit pronounced intrinsic gradients of microstructure and strain, which intensity scales inversely with the grain size and which decisively influence their materials functional properties. Therefore in order to understand structure-property relationship in polycrystals, it is necessary to characterize their inhomogeneous structural and mechanical properties. Scanning X-ray and neutron diffraction techniques operating with beam sizes from cm down to nm ranges are very convenient to resolve those gradients position-resolved deep in the material volume.

In this contribution, at first a very simplified introduction into the diffraction theory will be provided with an aim to underline especially information, which can be extracted from diffraction data. As a next, the scanning approaches will be demonstrated on some selected examples, like strain and microstructure mapping in thin films, steel tubes and surfaces, railway rails and wood.

Most of the examples are related to some industrial collaborations. The experiments were performed at large scale facilities in Grenoble (ESRF), Hamburg (Petra III), München (FRM II) and Berlin (BESSY).

Finally, the results should demonstrate that the diffraction represents not only a very powerful tool to analyse materials structure but can help us to design dedicated functional properties.

Why Neutrons?
A Compilation of Special Occasions

Erwin Jericha
Atominstitut, TU Wien, Austria
erwin.jericha@tuwien.ac.at

Neutrons are employed in the context of condensed matter studies in various situations. In general, it will turn out that they may not be the probes of first choice. However, there are conditions where their use is advantageous or they are even the sole option. In this presentation, we will discuss neutron studies with respect to the composition, the structure and the dynamics of materials, introduce the underlying principles and the relevant quantities and illustrate their application by exemplary research.

Tutorial on SAXS/WAXD with focus on soft matter and in-situ SAXS

Herwig Peterlik

Faculty of Physics, University of Vienna, Austria

herwig.peterlik@univie.ac.at

X-ray scattering is a powerful tool to identify structures at the nanoscale. The advantage of X-ray scattering – in comparison to concurrent or complementary techniques – is the simple sample preparation, the high resolution in reciprocal space and a good statistical accuracy of the collected data. The main advantage, however, is the possibility to look into materials under varying conditions by in-situ measurements, which enables to follow the evolution of nanostructures at different temperatures or loads, in different environments or under processing conditions.

An overview on SAXS/WAXD is presented and underpinned with selected examples from materials physics, materials chemistry and biological tissues.

Development and characterisation of advanced intermetallic titanium aluminide alloys by means of diffraction and scattering techniques

Petra Erdely[1], Peter Staron[2], Emad Maawad[2], Andreas Stark[2],
Helmut Clemens[1], Svea Mayer[1]
[1] *Department of Materials Science, Montanuniversität Leoben, Austria*
[2] *Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Germany*
petra.erdely@unileoben.ac.at

Intermetallic γ-TiAl based alloys are lightweight structural materials for challenging high-temperature applications in the aviation and automotive industry[1-4]. On account of their low density of roughly 4 $g \cdot cm^{-3}$, their high specific Young's modulus and strength at elevated temperatures, and excellent creep and oxidation properties, they have been implemented successfully, for example, as low-pressure turbine blades in environmentally-friendly aero engines[5,6]. During the last decades, the development of this class of alloys, which is relatively young compared with steels or Ni-base superalloys, has been promoted strongly by the employment of diffraction and scattering techniques based on both synchrotron X-rays and neutrons[3,7-9]. The useful complementarity of the two types of radiation is deeply engrained in the atomic structure of the intermetallic phases. In short, the consideration of the respective structure factors proposes the use of neutrons to study ordering phenomena based on strong superstructure reflections[8-11]. Unlike neutrons, X-rays yield strong ordinary and only weak superstructure reflections. As a result, they can be employed to address a variety of research topics, ranging from primal questions in the basic design of γ-TiAl based alloys over processing-related questions to application-relevant issues such as microstructural stability or the material's deformation behaviour under load[9]. In the present paper, three recent case studies are described, which demonstrate the applicability of diffraction and scattering techniques in the development of advanced γ-TiAl based alloys, and, thereby, suggest the ample opportunities these methods offer.

Intermetallic titanium aluminides suffer from a poor room temperature ductility[1,3]. One key to approach this issue may be found in establishing a thorough understanding of the deformation and load partitioning mechanisms at all scales. Especially in the case of multi-phase TiAl alloys such as the β-stabilised TNM alloy (Ti–43.5Al–4Nb–1Mo–0.1B (at.-%)), this understanding must be broadened to encompass all prevailing phases, whose role in the room temperature deformation is, partly, still the subject of much debate[1]. Case study one[12] shows how uniaxial tensile tests at a synchrotron radiation source were used to study the load-bearing mechanisms and their sequential order in a recent TNM sheet on the level of individual lattice planes and phases. Revealing four specific stages of deformation, the experiments disclosed that even though the TNM sheet succumbed macroscopically to brittle fracture, various mechanisms were indeed activated prior to failure. These mechanisms can be addressed to improve the room temperature ductility of the material.

In case study two[13] high-energy X-ray diffraction and small-angle X-ray scattering were combined with atom probe tomography as a direct imaging technique to explore opportunities for microstructural design in a ternary Ti–44Al–7Mo (at.-%) model alloy. In particular, the early growth stages of γ precipitates in a supersaturated β_o matrix were studied for the first time in an *in situ* manner, revealing options to optimise the proposed heat treatments that create refined microstructures with technologically promising features.

Both of the γ-TiAl based alloys mentioned above, i.e. the TNM and the Ti–44Al–7Mo (at.-%) alloy, can be classified as β-stabilised due to their elevated content of the β-stabilising elements Nb and Mo. While the body-centred cubic (bcc) β phase (A2 structure) facilitates hot working at high temperatures, its ordered low-temperature counterpart β_o (B2 structure) negatively influences the material's creep properties at service temperature (roughly 700 °C)[1-3]. As the properties of the alloys decisively depend on whether the bcc phase is present in its ordered or disordered form, methods of accurately determining the composition-dependent order/disorder transition temperature are of great importance. Case study three shows how neutrons[10,11] as well as today's highly brilliant synchrotron X-rays can conveniently be used in this regard, while conventional characterisation techniques reach their limits.

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20µm Cross-Sectional High-Energy Synchrotron X-Ray-Microdiffraction on Nitrided, Carburized and Shot-Peened Steels Reveals Near-Surface Microstructure, Phase and Stress Gradients

Sabine Bodner[1], Juraj Todt[2], Michael Meindlhumer[1], Emad Maawad[3], Norbert Schell[3], Jozef Keckes[1]
[1] Department of Materials Science, Montanuniversität Leoben, Austria
[2] Erich-Schmid-Institute for Materials Science, Austrian Academy of Sciences, Austria
[3] Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Germany~
sabine.bodner@unileoben.ac.at

The further development of thermo-chemical and mechanical surface processing techniques is just one part of the challenge to push the mechanical limits of existing high-performance steels – the second part is the design of appropriate characterization techniques to elucidate the impact of particular processing recipes on tool functional properties. The surface processing introduce usually complex near-surface chemical and/or mechanical gradients, which correlation to the overall tool performance is not yet understood.

In this work, high-energy synchrotron cross-sectional X-ray microdiffraction (CSmicroXRD) was used to characterize near-surface gradients of phases, microstructure and residual stresses at the cross-sections of nitrided, carburized and shot-peened martensitic steels. The experiments were performed at the HEMS beamline of the storage ring Petra III in Hamburg using the X-ray beam energy of 87.1 keV (wavelength of 14.24 pm). All samples were scanned with a stepwidth of 20 µm in transmission diffraction geometry using the X-ray beam with a cross-section of ~ (20x500) µm^2 (height x width) oriented parallel to the sample's surfaces. The sample's thicknesses along the beam were ~ 2 mm. The sample's microstructure and stress gradients were evaluated from the morphology and ellipticity of the Debye-Scherrer-rings using the PyFAI software package.

In the case of the nitrided sample (Fig. 1), the phase-analysis allowed to identify the interface between the diffusion zone of nitrogen and the martensitic matrix: the decarburization of the diffusion zone due to the interstitial diffusion of nitrogen led to the carbon enrichment at this interface at a depth of ~ 100 µm below the surface. Thus, the highest amount of retained austenite could be determined in this region. The stress analysis revealed the occurrence of two compressive stress maxima, each of about ~ -700 MPa (Fig. 1). The first maximum is located at the interface between the compound zone (rich on ε-nitrides) and the diffusion zone at a depth of ~ 30 µm below the surface. The position of the second compressive stress maximum can be directly correlated to the interface between the diffusion zone and the matrix material due to the internal carburization. This result is in good agreement with the complementary chemical depth profile analyses of nitrogen and carbon that were determined by energy-dispersive X-ray spectroscopy (EDX) and the microstructural information obtained by optical microscopy and scanning electron microscopy.

Within the carburized sample, the presence of retained austenite was identified up to the depth of ~ 400 µm below the surface. Maximum compressive stresses of ~ -320 MPa at a depth of 570 µm below the surface could be evaluated here in the martensite.

In the case of three shot-peened samples, CSmicroXRD was used to indicate the impact of different mechanical surface treatments on near-surface microstructure and stress states. The experiments revealed, that the different peening conditions influence the formation of compressive stresses, which depth's profiles differ in their peak values and the maxima positions. Depending on the peening procedure, one or two stress-maxima could be observed in the three samples.

In summary, this contribution demonstrates that high-energy CSmicroXRD provide unique high-resolved data on complex depth distributions of phases, microstructure and residual stresses, which are needed to gain deeper understanding of the influence of surface processing on the functional properties of high-performance steels. The high resolution of ~ 20 µm (and even less) obtained for truly bulk samples by using high energy photons is important, especially for all materials that exhibit non-linear and/or wavy gradients of microstructure, phases and stresses.

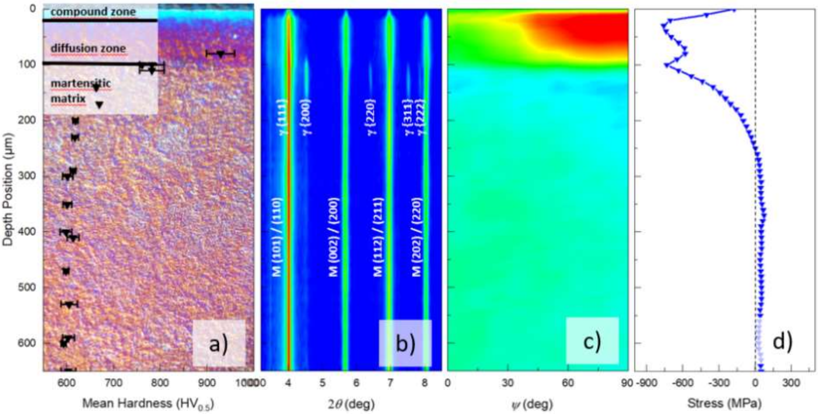

Figure 1: Exemplary results of the nitride sample. (a) Optical micrograph and hardness profile across the sample's near-surface region. (b) The phase plot indicates the presence of the martensitic matrix and a region with retained austenite at a depth of ~ 100 µm below the surface. (c) The analysis of the full-width at half maximum of the martensitic (101)/(110) peak reveals the influence of the nitrogen diffusion on the microstructural properties. (d) The stress depth profile indicates the presence of two stress maxima which occurrences correlate with the chemical and phase gradients.

Stress and Microstructure Distributions across Scratch Track Cross-Sections in a CrN-Cr bilayer on Steel Revealed by 50nm X-ray Diffraction.

Michael Meindlhumer[1,2], J. Todt[2], M. Rosenthal[3] C. Mitterer[1],
R. Daniel[1], J. Keckes[1,2]
[1] *Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at the Department of Materials Science, Montanuniversität Leoben, Leoben, Austria*
[2]*Department of Materials Science, Montanuniversität Leoben and Erich Schmid Institute for Materials Science, Austrian Academy of Sciences, Leoben, Austria*
[3]*ESRF Grenoble,*
michael.meindlhumer@unileoben.ac.at

Scratch tests are widely performed to evaluate the adhesion strength of brittle films to the substrate[1], but up to now, there is no experimental study which focusses on characterization of nanoscale stress distributions generated in the film. Cross-sectional X-ray nanodiffraction[2] has been proven as a powerful tool to investigate ex-situ stress gradients generated in films due to the indentation[3] and is now applied for the ex-situ investigation of a CrN/Cr thin film after scratch tests.

Two tests with the particular applied loads of 200 mN and 400 mN were performed on a magnetron sputtered multi-layered thin film consisting of 1.2 and 2 µm thick CrN and Cr sublayers, respectively, deposited on a high-speed-steel substrate. Subsequently, a cross-sectional lamella with a thickness of ~20 µm was cut out and polished from the sample. The synchrotron cross-sectional X-ray nanodiffraction measurements were carried with a beam of 50 nm x 50 nm (oriented parallel to the track directions) to resolve changes in multi-axial stress fields and microstructure within scratch track areas. Additionally a 3D finite element simulation was performed and correlated to the synchrotron results.

Two cross-sections prepared by focused-ion-beam (FIB) milling revealed (i) columnar grain microstructure in both films sublayers, (ii) grain reorientation and plastic deformation and (iii) no significant cracking in the scratch track area formed by a load of 200 mN, whereas (iv) multiple cracking in the top CrN sublayer after the scratching with 400 mN. Texture analyses carried out for the CrN 200 and the Cr 110 peaks along the critical lines throughout the scratches revealed bending of the columnar grains up to the angle of 30° from the out-of-plane orientation, being consistent with the cross-sectional morphologies observed by scanning electron microscopy (SEM).

In the as-deposited multilayer, residual stresses of ~−3 and −1 GPa in the CrN and Cr sublayers, respectively, were found. After the scratching complex in-plane stress distributions were found in the CrN sublayer reaching the magnitude of ~-6 GPa in the near-interface region, whereas residual stresses were nearly relaxed next to the surface cracks. In the out-of-plane direction compressive stresses of ~500 MPa were found directly under the scratch path for the scratch carried out with the smaller load, whereas ~2 GPa compressive stresses were found for the scratch performed with the 400 mN load. Within the Cr sublayer, tensile in-plane stresses were found near the CrN-Cr interface regions and compressive stresses of ~-1.5 GPa were evaluated near the film-substrate-interface. Complex, antisymmetric shear stress distributions were found in both sublayers and in both samples, which are in good agreement with the results from the three-dimensional finite element model.

The ductile Cr sublayers therefore function as a stabilizing component for the CrN, which allows a bending of the CrN sublayer, whereas above a critical loading, the mechanical integrity of the CrN is disrupted and the indenter presses the CrN sublayer into the softer Cr sublayer.

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Hexagonal Si-Ge, characterization of a new crystalline phase realized in nanowire structures

D. Ziss[1], M. Watzinger[1], E. Fadaly[2], E. P. A. M. Bakkers[2], J. Stangl[1]
[1] *Inst. of Semiconductor and Solid State Physics, JKU Linz, Austria*
[2] *Dept. of Applied Physics, Eindhoven University of Technology, The Netherlands*
dorian.ziss@jku.at

Semiconductor-devices for electronic applications are nowadays mainly based on silicon and germanium. These elements natively crystalize in the cubic crystal structure and are known to be indirect band-gap semiconductors. Hence, efficient light emission was not realized so far and achieving lasing from group IV elements has been the "holy grail" in silicon technology for decades. When changing the crystal structure, from the native cubic to the hexagonal (hex.) phase, a direct band-gap can be realized for Ge-rich, hex-$Si_{1-x}Ge_x$ alloys.

Theoretical calculations[1] and recent measurements already have proven an efficient light emission with a tuneable bandgap (1.7 µm to 4.1 µm), depending on the Ge concentration, for hex. $Si_{1-x}Ge_x$ These alloys could first be synthesised using nanowire (NW) structures as epitaxial templates for hexagonal growth.

We present methods how to determine the lattice parameter of such $Si_{1-x}Ge_x$ alloys in the hex. crystal phase from x-ray diffraction experiments in combination with finite-element-modelling (FEM) simulations.

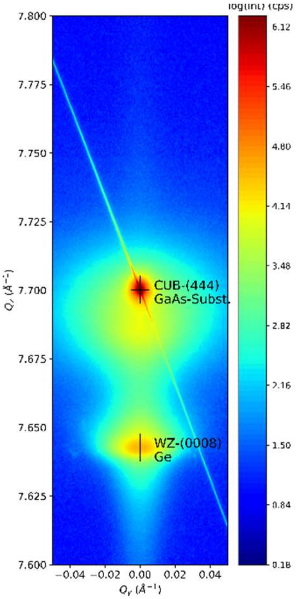

Figure 1 - RSM of the GaAs (444) substrate reflection and the hex. (0008) Ge reflection measured for a wire-ensemble with pure Ge in the outer shell.

Hexagonal $Si_{1-x}Ge_x$ with variable Ge-contents has first been realized in a rather complex core-shell-shell NW system grown on pre-patterned GaP (111) substrates[2]. The core material is hex. GaP, followed by an intermediate Si-shell and the outermost $Si_{1-x}Ge_x$ shell. The core-shell-shell structure of the NWs is needed to reduce the lattice mismatches between the different materials which is especially relevant for higher Ge-concentrations. We employed focused synchrotron radiation to measure reciprocal space maps of substrate and NW Bragg reflections. The measured Bragg reflections contain collective information on the hexagonal system including the strain induced in the different core-shell-shell materials. Hence, further in-depth data treatment is necessary to extract information about the lattice parameter of the un-strained materials. For this purpose, the X-ray diffraction measurements are compared to FEM simulations (on the strain present in the system) in combination with a successive calculation of the expected diffraction patterns of the strained wires. The calculated diffraction patterns contain information on the shape and intensity distribution of different Bragg peaks which are then compared to the measured peaks. If the FEM simulations of the wire model accurately reproduced the intrinsic strain distribution in the real-NW, the unstained hex. lattice constants of $Si_{1-x}Ge_x$ can be extracted.

However, for very high Ge-concentration up to pure Ge the accumulation of strain in the core-shell-shell system is too high and the formation of defects and plastic relaxation is expected. Hence, a second set of NW-samples where the core material is hex. GaAs (grown on GaAs (111) substrates) directly followed by the $Si_{1-x}Ge_x$ -shell has been successfully characterized. Using hex. GaAs as core material allows for the first time the growth of large-scale, pure hex. Ge due to the low lattice mismatch to the hex. GaAs (<0.1%). This system enables a direct characterization of this rather exotic crystal phase observed in Ge, via X-ray diffraction. As an example, a reciprocal space map of a GaAs- Ge NW ensemble measured at DESY (P08) is shown in Fig. 1.

In the presented work we could successfully characterize the lattice parameters of hex. $Si_{1-x}Ge_x$ alloys for various Ge concentrations, up to pure Ge. The investigated structures are highly promising for further developments in the silicon industry, e.g. towards the realization of optical circuits for inter-chip connections. Hence, this work can be seen as one step towards this aim, since the knowledge of the lattice parameters allows highly accurate calculations of the band-structure as of function of the Ge content in $Si_{1-x}Ge_x$ alloys needed to engineer the band structure towards the desired emission properties.

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Synchrotron radiation is a powerful tool to study supercapacitor charging behaviour

Christian Koczwara[1], Christian Prehal[2], Oskar Paris[1]
[1] *Institute of Physics, Montanuniversität Leoben, Austria*
[2] *Institute for Chemistry and Technology of Materials, Graz University of Technology, Austria*
christian.koczwara@unileoben.ac.at

Electrical double layer capacitors (EDLCs) or supercapacitors are high power density energy storage devices with long cycle lives. They play an important role in applications demanding for short charging and discharging times. EDLCs typically consist of two nanoporous carbon electrodes and a glass fibre separator, all soaked with a liquid electrolyte. Applying a voltage between the electrodes leads to the formation of an electrical double-layer at each electrode-electrolyte interface, causing the capacitive behaviour. The use of spectroscopic in situ techniques increased the qualitative understanding of ion charge storage mechanisms over the last few years, yet details remain unanswered. Recently, we have shown that in situ small-angle X-ray scattering (SAXS) can be used to study global ion fluxes as well as local ion rearrangement in disordered nanoporous carbon materials.[1]

Here, we report on the extension of these studies to hierarchically ordered pore systems. We investigated two hierarchically organized carbon materials with comparable macroporosity, but inverse mesopore structures consisting of cylindrical mesopores or cylindrical carbon nanorods, respectively, arranged on a 2D hexagonal lattice. Additionally, micropores were introduced into both systems by CO_2 activation. By combining structural characterization and electrochemical measurements, we were able to clarify the influence of the pore structure on the rate handling stability for high charging and discharging rates. Moreover, tracking the Bragg peaks from the ordered mesostructure during charging and discharging allowed us to measure electrosorption-induced deformation of the electrode material on the nanometer scale along with in situ electrochemical dilatometry.[2]

Following the X-ray transmission signal as a function of the applied voltage turned out to be a very powerful method to study global ion concentration changes while operating the supercapacitor cell. We found that the charging behaviour is influenced by different parameters like scan rate, salt concentration and the cell design. With decreasing salt concentration and decreasing scan rate we found that the charging mechanism changes from ion replacement to counter-ion adsorption. This finding is of considerable practical relevance when comparing results obtained from different in situ techniques such as in situ SAXS or in situ NMR.[3] However, the transmission signal stems from the concentration changes of both ion types, requiring additional information to separate them. Recently, we have for the first time explored the element specific (i.e. ion specific) analysis using anomalous small-angle X-ray scattering (ASAXS), which can help to gain deeper understanding of the behaviour of individual ion types in an operating EDLC cell.

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Smart neutrons for in-situ und operando research in the field of batteries and high-temperature alloys

Ralph Gilles
TU München, Forschungs-Neutronenquelle Heinz Maier-Leibnitz
Lichtenbergstr. 1, 85747 Garching near München

ralph.gilles@frm2.tum.de

This contribution gives an overview how neutrons with their unique properties contribute in the development of new battery cells. During charging and discharging of NMC/graphite cells the intercalation of Li in the graphite layers can be observed in situ with neutron diffraction (ND) as such measurements are sensitive to detect LiC_x phases as LiC_6 and LiC_{12} during the intercalation/de-intercalation process[1]. Under fast charging conditions and low temperatures the appearance of Li plating can be studied. On larger scales of >50 micrometer neutron imaging (radiography and tomography) enables a non-destructive view inside the cell to make visible for example how the electrolyte filling with the distribution of the electrolyte in the cell between the layer stacks in a pouch cell takes place[2]. The use of neutron induced prompt gamma activation analysis (PGAA) is a powerful tool to describe the capacity loss of the cell caused by tiny metal deposition on the graphite anode after charging/discharging processes[3]. The method of neutron depth profiling (NDP) is suited to study near surface phenomena as the Li distribution in electrodes[4].

Neutron scattering is a powerful tool to study Ni-base superalloys and new alloys beyond this type of alloy. Size distributions, morphology and volume fraction can be characterized in situ at high temperatures in real bulk samples. Information on precipitate dissolution and formation can be carried out as well. Examples using neutron diffraction and small-angle neutron scattering will be presented[5,6].

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Grazing Incidence X-ray Scattering Techniques:

Crystallisation of Molecules at Solid Surfaces

Roland Resel
Institute of Solid State Physics, Graz University of Technology, Austria

roland.resel@tugraz.at

X-ray scattering techniques are widely used to characterise the structural properties of organic materials in terms of their crystalline and morphological features like type of phase, molecular packing, preferred orientation of molecules, crystallinity, etc. In case of organic thin films specific experimental techniques are applied. The tutorial talk will introduce x-ray scattering techniques using grazing incidence conditions of the primary beam, namely x-ray reflectivity and grazing incidence x-ray scattering. The fundamental principle of these two methods will be discussed in terms of the optical properties of x-rays, a short comparison to the analogous techniques using neutron beams will be drawn. The experimental possibilities of these methods will be introduced on basis of thin films of benzthieno-benzthiophene type molecules. The crystallisation starting from sub-monolayer films up to thick films will be introduced. Special emphasis will be drawn on the ability of the molecules to form polymorph crystal structures within thin films. And the techniques to solve crystal structures from thin films will be discussed.

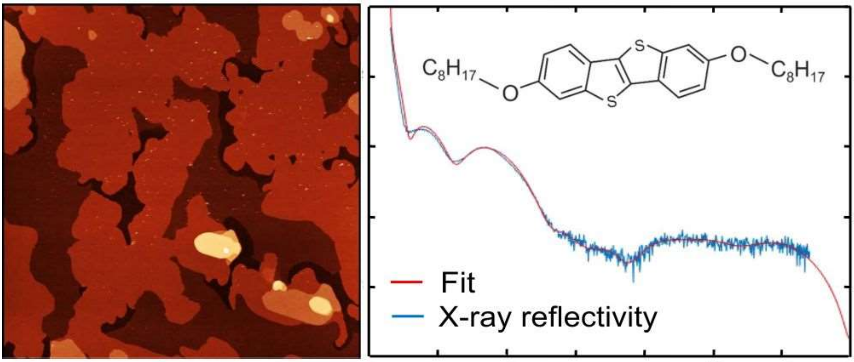

Figure: Atomic force microscopy image of a thin film of the molecule dioctyloxy-benzothienobenzthiophene with a nominal thickness of 1.5 monolayer (left part); X-ray reflectivity curve and fit of the experimental data to reveal the electron density distribution (right part).

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Diindenoperylene Molecules on 2D MoS$_2$ Substrates

Nada Mrkyvkova[1], Jakub Hagara[1], Martin Hodas[2], Peter Nádaždy[1], Ashin Shaji[1],
Eva Majková[1], Matej Jergel[1], Michaela Sojková[3], Martin Hulman[3], Peter Siffalovic[1],
Frank Schreiber[2]
[1] *Institute of Physics, Slovak Academy of Sciences, Slovakia*
[2] *Institute of Applied Physics, University of Tubingen, Germany*
[3] *Institute of Electrical Engeneering, Slovak Academy of Sciences, Slovakia*
nada.mrkyvkova@savba.sk

Recently developed one-zone sulfurization technique[1] enables fabrication of uniform, large-area few-layer MoS$_2$ films with a high degree of crystallinity. In this contribution, we show that the crystallographic orientation of MoS$_2$ *c*-axis is directly influenced by the layer thickness and/or sulfurization temperature. To be more specific, the *c*-axis varies from the out-of-plane to in-plane orientation for samples with the MoS$_2$ thickness of 3 and 9 nm, respectively. Furthermore, we found out that the *c*-axis orientation subsequently controls the orientation of small organic molecules, such as diindenoperylene (DIP), grown on the top of MoS$_2$ layer. Employing the grazing-incidence wide-angle X-ray scattering (GIWAXS), we observed that the DIP molecules are oriented in the lying-down configuration when the *c*-axis is normal to the substrate plane and adopt the standing-up orientation when the *c*-axis is oriented parallel to the substrate plane, see Figure 1. To the best of our knowledge, the molecular orientation was so far manipulated exclusively by selecting an appropriate substrate[2], but a different molecular orientation on the same kind of substrate was not observed up to now. This result has direct implications for a tailored preparation of small-molecule organic films for diverse applications in (opto)electronics.

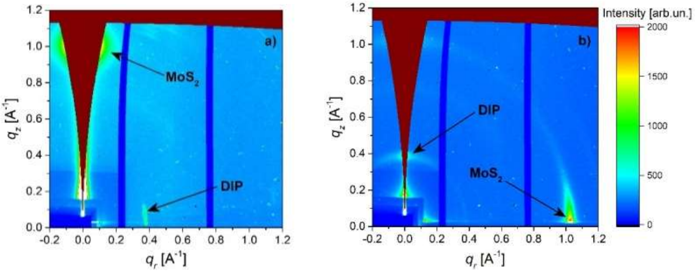

Figure 1: Reciprocal space map shows 002 diffraction peak, coming from the MoS$_2$ layer with a thickness of a) 3 nm and b) 9 nm, and 001 reflection of the 13 nm thick layer of DIP.

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The Conundrum of Organic Dye-Silica Hybrids: How Can Neutrons and Synchrotron Radiation Techniques Assist Us?

Hipassia M. Moura[1,2] *Herwig Peterlik*[3]*and Miriam M. Unterlass*[1,2,4*]

[1] *Institute of Materials Chemistry, TUWien, Vienna, Austria*
[2] *Institute of Applied Synthetic Chemistry, TUWien, Vienna, Austria*
[3] *Faculty of Physics, University of Vienna, Vienna, Austria*
[4] *CeMM Research Center for Molecular Medicine of the Austrian Academy of Sciences, Vienna, Austria*
*miriam.unterlass@tuwien.ac.at

Perylene-diimide dyes (**Fig. 1a**) are widely used as pigments, fluorescent sensors, and n-type semiconductors in organic electronic because of their high photostability, interesting optical and electronic properties.[1,2] A few supramolecular architectures of conjugated dyes with unique optical properties have been reported in the literature[3,4]. In the present contribution, we report the preparation of silica nanoparticles (**Fig. 1b**) by incorporating rylene-dyes inside the silica matrix (**Fig. 1c**) using one-pot and one-step hydrothermal synthesis (HTS).

HTS is a classical inorganic method used to generate various inorganic compounds such as gems and zeolites that is environmentally friendly. Recently[5,6] we showed that organic high-performance dyes can also be prepared by HTS. However, the simultaneous synthesis of both components, organic dye and inorganic SiO_2, remains unexplored.

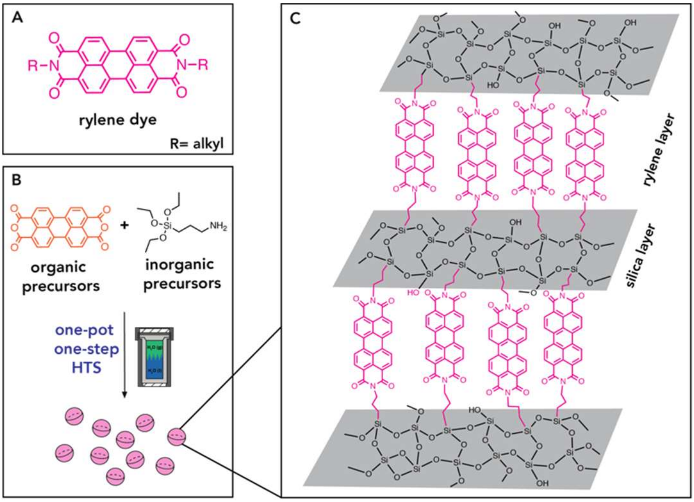

Figure 1. Schematic model of perylene-incorporated silica nanoparticles prepared by hydrothermal synthesis. A) Example of perylene-diimide dye. B) One-pot, one-step HTS of dye@SiO_2. C) Layered assembly of dye@SiO_2, containing a perylene rich region and silica region.

Since both components require the same hydrothermal regime for their synthesis, the preparation of supramolecular structures like dye@SiO_2 is feasible. Preliminary data suggests that the dye@SiO_2 hybrids obtained by one-pot HTS consist of silica nanoparticles, which form perylene rich regions assembled together with layered silica regions (**Fig. 1c**).

Here, we address important questions about the construction of the dye@silica: *(i)* the aggregation and/or segregation of rylene molecules inside and outside the silica nanoparticles, *(ii)* chemical and electronic interactions between the rylene molecules and *(iii)* growth of the SiO_2 nanoparticles and organization of the organic moieties inside the inorganic SiO_2 lattice. Such control on arrangement and distribution of the molecules inside the silica lattice is expected to show interesting optical properties.

Neutrons and synchrotron radiation are unique probes for the characterization of structures in materials, including *e.g.* the atomic structure, density and size distribution of pores/galleries, roughness and correlation of internal and external interfaces of dye@SiO_2 hybrid materials. Structure and morphology of the hybrid materials here exploited have been characterized using a range of techniques such as electron microscopy, spectroscopic measurements and thermal analysis. However, more information regarding the structures' formation need to be exploited.

To date, the hydrothermal crystallization of an organic or organic-inorganic hybrid material have not been studied in real time. *In-situ* studies of hydrothermal crystallization via neutron diffraction (ND), for example, would be an excellent technique to identify *(i)* the rates of consumption of starting compounds and appearance of products, *(ii)* the course of crystallization in terms of crystallization rates, *(iii)* crystalline intermediates and *(iv)* give insights on the mechanistic hypotheses of crystallization of organic and inorganic crystalline phases during hydrothermal synthesis of the dye@SiO_2 hybrid materials. Such measurements have been performed by O'Hare *et. al.*[7] to study the hydrothermal zeolite formation at the Rutherford Appleton Laboratory (UK), and it can indeed be expanded to the detailed understanding of HTS mechanisms of dye@SiO_2 hybrids preparation.

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X-ray Spectroscopy - a Tool for Material Testing

Peter Kregsamer

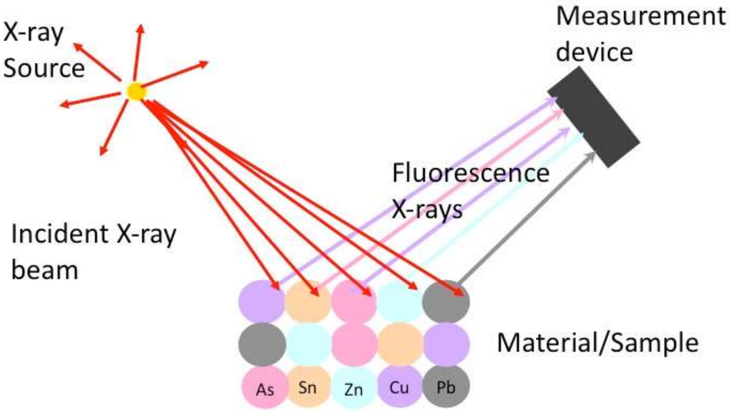
Atominstitut, TU Wien, Austria

pkregsam@ati.ac.at

For the energy range from 1 – 100 keV absorption of X-rays is the dominant effect. The consequent emission of characteristic X-rays (X-ray fluorescence) and their detection is used in X-ray fluorescence spectroscopy (XRF). Elements of the periodic table from Na upwards to the heaviest ones can be detected. Quantitative results for samples containing various elements in the range from 100% down to 1 ppm can be obtained routinely. In principle the technique is non-destructive, the sample can be returned to the customer in the original state. Otherwise a well-selected specimen will be used for the analysis.

Several technological developments can extend the detectable elements' range, the lower limits of detection can be brought down by orders of magnitude, and imaging techniques can be applied as well as chemical speciation.

The fundamentals of XRF and limiting influence factors will be discussed and selected advanced applications presented.



Facilitating synchrotron radiation for drug development

Oliver Werzer

Institute of Pharmaceutical Sciences, Dept. Pharm. Technology
University of Graz, Austria

oliver.werzer@uni-graz.at

The development of medication for effective therapy is complex. From the identification of a chemical lead to synthesis of molecules, preformulation studies, upscaling, various clinical studies or drug formulation. In many of these stages, synchrotron radiation can be facilitated to gain important information on the properties of the drugs, excipients and their combinations. In this contribution, an overview on various experimental techniques typical present at synchrotron sources is provided and how their (potential) use for various aspects in the pharmaceutical sciences can be facilitated to develop future medications.

In Situ GISAXS Investigation of PtNi Bimetallic Alloy Under Operational Conditions of Fuel Cells.

Marco Bogar[1,2], Ivan Khalakhan[3], Roman Fiala[3], Josef Mysliveček[3,]
Vladimir Matolin[3], Heinz Amenitsch[2]

[1] *CERIC-ERIC*
[2] *Institute of Inorganic Chemistry, Graz University of Technology, Austria*
[3] *Faculty of Mathematics and Physics, Charles University, Prague, Chzech Republic*
marco.bogar@ceric-eric.eu

In the last years, several Platinum-based bimetallic alloys have been studied as possible substituents of pure Platinum catalysts in Proton Exchange Membrane Fuel Cells (PEMFCs) to strongly reduce production cost. In particular, PtNi alloy was found to be a promising alternative to pure Pt due to its very good mass activity[1] and its competitive catalytic activity[2]. Despite these advantages, the acid-based environment in which catalyst has to operate, causes the dissolution of the less noble metal and the consequent degradation of the catalyst layer. In this picture, in situ Grazing Incidence Small Angle X-ray Scattering combined with electrochemistry is used to investigate the real-time kinetics of the surface degradation of PtNi alloy during electrochemical cycles. The study has been performed at different conditions, which simulate the real fuel cell operation. Obtained results, complemented with in situ Electro-Chemical AFM, have been used to better describe the degradation of the aforementioned catalyst and to reveal in depth the structural and compositional changes during fuel cell operation.

The authors acknowledge the financial support from project CEROP, which is a CERIC-ERIC internal research project.

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Bio-Inspired Band Gap Engineering of Hybrid Perovskite $MAPbBr_3$

Arad Lang[1], Boaz Pokroy[1]
[1] *Department of Materials Science and Engineering,*
Technion – Israel Institute of Technology
aradlang@campus.technion.ac.il

One of nature's strategies for enhancing the mechanical properties of biominerals is the incorporation of organic molecules[1]. As was shown for calcite, incorporation of certain individual amino acids is possible synthetically[2]. Moreover, this incorporation enables fine tuning of calcite's hardness, as the organic molecules induce internal stresses[3].

Recently, a novel bio-inspired band gap engineering technique was developed in our lab, based on this phenomenon. It was shown that amino acids can get incorporated inside ZnO lattice, and induce an increase in its optical band gap[4,5]. This change in band gap occurs due to the creation of a superlattice, in which the amino acids acts as potential barriers for the charge carriers[6].

In this research, we try to extend the knowledge about the effect of incorporated amino acids into different crystals. We focus on $MAPbBr_3$ – a visible-light semiconductor hybrid organic-inorganic perovskite. We found that Lysine, having an NH_3^+ group (similar to the MA^+ anions in the crystal) is the amino acid which incorporates the most. It induces a shrinkage of the unit cell, together with changes in the crystals morphology. Additionally, the incorporation of Lysine increase the optical band gap of $MAPbBr_3$ up to 1.2%.

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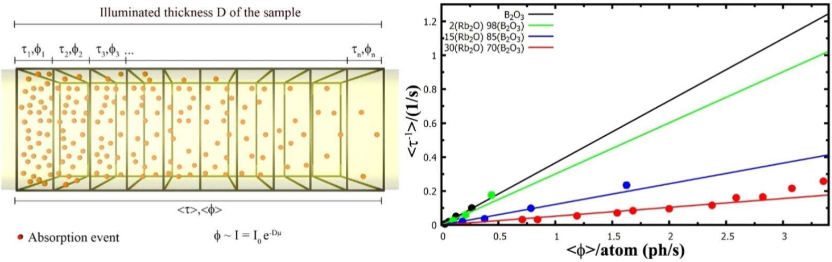
Studying Beam-induced Dynamics in Amorphous Ionic Conductors

<u>K. Holzweber</u>[1], C. Tietz[1], T. Fritz[1], M. Leitner[2], J. Dabrowa[3], B. Sepiol[1]
[1] Faculty of Physics, University of Vienna, Boltzmanngasse 5, 1090 Vienna
[2] Technical University Munich, Lichtenbergstraße 1, 85747 Gaching
[3]AGH University of Science and Technology, 30-059 Krakow, Poland
katharina.holzweber@univie.ac.at

By collecting series of diffuse scattering patterns with coherent X-rays aXPCS (atomic-scale X-ray Photon Correlation Spectroscopy) has become a very powerful technique to probe atomic motion in crystalline and in amorphous materials. The resulting "motion picture" of speckles provides information on the atomic dynamics like the decay time τ or the diffusion coefficient of the matter. Ruta et al.[1] showed, however, that the atomic diffusion in glasses is not only dependent on the temperature and the scattering vector q, but also on the flux of the coherent beam, which could be seen as a great opportunity to indirectly probe physical properties of materials like e.g. bonding features. By measuring different kinds of alkali borate and silicate glasses with different alkali concentrations we could gain information about correlations between the beam-driven dynamics and the physical properties of these ionic conductors.



<u>Figure 1</u>: Left: sketch of a beam-induced dynamics behavior, where τ_i and ϕ_i (i=1,2,3,..,n) are the correlation times and fluxes in every ΔD of the illuminated part of the sample. Right: flux-dependency for $x(Rb_2O)\ (1-x)B_2O_3$ glass with x= 0, 0.02, 0.15 and 0.30.

By measuring the diffusion in B_2O_3 within a wide q-range we suggest that the beam causes a liquid-like state of the illuminated volume in insulators, whereas in ionic conductors other diffusion mechanisms like atomic jump processes emerge [2,3].

This work was financially supported by the Austrian Science Fund (FWF) project P-28232.

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Contrast variation in Small-Angle X-ray Scattering for Material Science Application

Sylvio Haas
DESY, Photon Science Division, Hamburg, Germany
sylvio.haas@desy.de

Modern applications and basic research in catalysis, biotechnology and materials are often concerned with hybrid and synergetic systems. In other words, systems that brings enhanced properties and performances due to their internal complexity. The challenge is now in understanding the interactions leading to their assembly and operation. Due to their inherent chemical and structural complexities a combination of several techniques simultaneously or an advanced method is necessary to determine structural and chemical information of materials.

Small-Angle X-ray scattering (SAXS) as well as Small-Angle neutron scattering (SANS) are well established methods to investigate nanostructured materials. Information about particle size distribution, particle shapes, and inter-particle interaction potentials can be determined. In neutron scattering the contrast between different phases can be manipulated by replacing partially Hydrogen atoms with Deuterium atoms. This contrast variation technique in case of neutrons helps to disentangle complex nanostructures. In case of X-ray scattering another concept can be used to manipulate the scattering contrast. This is called anomalous scattering. In addition to the structural information determined from SAXS chemical information about the different phases can be evaluated by different methods from the anomalous scattering behaviour.

Within this contribution the basic concept of anomalous scattering and the different analysis techniques will be presented and discussed using selected examples:

- Ruthenium – Selenium based catalyst for fuel cell applications[1]
- Gold – Palladium catalyst for chemical conversion applications[2]
- Complex glass-ceramic that shows a high frequency up-conversion efficiency[3]

Furthermore, the new upcoming SAXS beamline (P62) at PETRA III, DESY will be presented. This new beamline is dedicated for anomalous SAXS as well as SAXS computed tomography experiments especially for material science research.

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Micro-/Nano-beam X-ray Diffraction & Scattering

Manfred Burghamer
ESRF, Grenoble, France
burgham@esrf.fr

Since more than two decades the Microfocus Beamline (ID13 ESRF) is dedicated to employing monochromatic X-ray micro- and nano-beams for diffraction and scattering experiments which are carried out in a spatial raster scanning modes or by recording diffraction time series on microscopic samples. Typical focal spot sizes are ranging from 2 microns down to 30 nm in diameter. While maintaining enough flexibility to accommodate an ample variety of sample environments, diffraction geometries, and complementary methods such as x-ray fluorescence analysis, the beamline's scientific program in terms of user and inhouse research covers also a wide range of topics. Where the main focus is on soft matter studies and life science with special emphasis in bio-materials, there is also a considerable fraction of hard condensed matter research. In this presentation we will present the basics of the related methodology illustrated by selected examples ranging from high resolution fibre diffraction to millisecond in-situ chip calorimetry making use of the 750 kHy frame-rate of the beamline's pixel array detector (EIGER 4M). We will conclude with an outlook on future opportunities regarding the increased performance to be expected from the ESRF-EBS upgrade project. Specific challenges such as managing radiation damage effects and the high volume data rates certainly emerging from a considerable increase in data acquisition speed will be discussed**.**

Extreme condition research using synchrotron radiation: Extended stability of carbon dioxide above 1 Mbar

Ronald Miletich

Institut für Mineralogie und Kristallographie, Universität Wien, Austria

ronald.miletich-pawliczek@univie.ac.at

X-ray crystallography under extreme and non-ambient conditions is on of the rising fields in modern material-related sciences related to investigations on solids. It is the rising demand for reaching non-ambient conditions and crossing supposedly challenging limits, and implementing novel techniques available for new experimental setups in order to reach the supposedly in accessible conditions. The motivation is mostly given by questions originating from the missing knowledge about the interior of planets, the state of matter in exoplanets and other astrophysical bodies, or the role of materials in shock conditions such as impacts. Diffraction is one of the methods that provides insight to the structure of crystalline solids from atomic point of view, especially exemplified for carbon dioxide.

In this tutorial the techniques used for the application of high and extreme pressure conditions in combination with temperature variations for in-situ X-ray diffraction will be illuminated. The instrumentation of large-volume pressures and diamond-anvil cell techniques will be explained, and particular attention is payed to the optimization of experiments using conventional laboratory X-ray radiation sources, synchrotron beamlines, and the possibilities for the future use of free electron lasers.

Introduction to X-ray Tomography

John W.C. Dunlop
Chemistry and Physics of Materials, Univ. Salzburg, Austria

john.dunlop@sbg.ac.at

Temperature- and Water Triggered Seed Release of Banksias

Michaela Eder

Max-Planck-Institute of Colloids and Interfaces, Department of Biomaterials, Potsdam, Germany

michaela.eder@mpikg.mpg.de

Fire is a component of the natural Australian landscape since millions of years. As a consequence, vegetation has adapted to these natural "harsh" conditions with some plants developing strategies for re-establishment after fire, eg resprouting and/or germination from seeds. In many cases seeds are accumulated between fires and stored in the soil or in the canopy. Species of the genus Banksia are a prominent example for canopy seed storage. However, seed storage periods are species dependent and seed release may occur upon maturity in some species whereas others rely on elevated temperatures, eg during fires. Even within one species both strategies and transitions between them are possible.

To explore the underlying mechanisms for timed seed release we investigated *Banksia attenuata*, a species known for both spontaneous and fire-triggered seed release. The seed pods (follicles) which contain two winged seeds and a separator in between develop on infructescences (see Figure) after pollination of a showy spike which contains several thousand flowers. We collected infructescences with mature follicles without metabolism along the west Australian coast line from Perth to Eneabba (approximately 250 km, black dots in the map in Figure, green shows distribution range of *B. attenuata*). Infructescences from the southern sampling sites contained many open follicles, whereas most of the follicles from the northern sites remained closed (see Figure). Interestingly, follicle opening temperatures of the collected samples changed gradually from 54°C in the south to 72°C in the north[1]. Detailed analyses of the follicle material were performed with various methods (eg. light- and electron microscopy, WAXD, µCT, spectroscopy, …). The investigations revealed material composition and structure providing seed pod stability for up to 15 years[1,2]. To understand the temperature-triggered opening heating experiments in a µCT scanner were performed with the aim to detect the area of initial opening. Based on this data we were furthermore able to show that temperature-triggered follicle opening is controlled by internal curvature.

However, temperature-triggered initial opening is not sufficient for seed release. The seed pods need water exposure (eg. rain) for further opening. Again µCT experiments are a powerful tool to track water-fuelled movements of the seed pod.

Our findings do not only provide inspiration for the development of simple “self-moving” or “self-sensing” devices but have direct implications for plant conservation in fire-prone ecosystems.

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Determining the Microstructural Characteristics in Paper-based Materials from X-ray Tomography

Eduardo Machado-Charry[1,2], *Matthias Neumann[3], Volker Schmidt[3], Robert Schennach,[1,2], Karin Zojer[1,2]*

[1] *Christian Doppler Laboratory for mass transport through paper, Graz University of Technology, Graz, Austria*
[2] *Institute of Solid State Physics and NAWI, Graz University of Technology, Graz, Austria*
[3] *Institute of Stochastics, University, Ulm, Germany*
machadocharry@tugraz.at

X-ray microcomputer tomography (X-µCT) has become a crucial tool for characterizing porous materials. More specifically, many X-µCT experiments seek to determine the complex microstructure of these materials and represent it with a set of parameters, e.g., the porosity and the tortuosity. Both parameters critically determine properties of paper-based materials, like the air permeance. Linking such global paper properties to the microstructure is of paramount importance for paper industry.

The microstructural parameters of paper samples have been successfully extracted using X-µCT characterizations both with synchrotron radiation[1,2] facilities and desktop devices[3]. However, given that these parameter extractions were limited to a single sample for each type of paper, the question arises whether the 3D pore structure of a single sample is enough to capture the nature of a whole paper sheet. Paper consists of a complex fibre network and is inhomogeneous on multiple scales; the latter cause inherent structural variations at length scales that may not be capture due to the limited size of the sample.

In this contribution we aim at necessary steps to surpass limitations in the scanned area. In first step, we present recent experimental results acquired at TOMCAT-SLS. An optimized setup allowed us to scan an area of approximately 270 mm^2 at a pixel resolution of 0.65 µm. This area exceeds the previously achieved sample areas of 2 mm^2. In a second step, we will present our route to determine the statistical correlation between porosity and tortuosity from the microstructure. This statistical analysis aims at crafting a parametric model, that allows us to compute the distribution of the local mean geodesic tortuosity from the distribution of local porosity for a given paper sample.

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Utilizing Synchrotron Radiation to Reveal the Structure-function Relation in Biogenic and Bioinspired Materials

Boaz Pokroy
Technion Haifa, Israel
bpokroy@technion.ac.il

In-situ / Pump-probe Experiments at the Austrian SAXS beamline

H. Amenitsch
Institute of Inorganic Chemistry, Graz University of Technology, Austria
amenitsch@tugraz.at

By probing the structural regime of 1 to 100 nm Small Angle Scattering is the perfect tool to investigate hierarchical self-assembly being a fundamental principle in nature with astonishing supramolecular architectures. Therefore, self-assembly of biostructures inspired many innovative materials in nanotechnology. A key issue in such studies is the fast and controlled perturbation of the system under realistic conditions and environments, in which small angle scattering is in particular suited due to the accessibility of the sample. In general, solids, liquids, films and aerosols can be investigated, therefore covering all states of matter.

This presentation should highlight the current status of the available instrumentation in the field of in-situ/pump-probe experiments and exemplify the research facilitated by this methods.

X-ray magnetic circular dichroism: basic principles and recent developments in time-resolved imaging

Andreas Ney
Solid State Physics Division, JKU Linz, Austria
andreas.ney@jku.at

Physics with Neutrons

Hartmut Abele
Atominstitut, TU Wien, Austria
hartmut.abele@tuwien.ac.at

Deformation and Crystallization Kinetics of a Soft Magnetic Iron Based Bulk Metallic Glass

Monika Antoni[1,2], Reinhard Pippan[1,3], Baran Sarac[3], Sergey Ketov[3],
Andrea Bachmaier[3], Christian Polak[4], Florian Spieckermann[1],
Michael Zehetbauer[5], Roland Größinger[2], Jürgen Eckert[1,3]

[1]Chair of Materials Physics, Montanuniversität Leoben, Austria,
email: monika.antoni@unileoben.ac.at
[2]Institute of Solid State Physics, Vienna University of Technology, Austria
[3]Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Austria
[4]Vacuumschmelze GmbH & Co. KG (VAC) Rapid Soldification Technology, Germany
[5]Research Group Physics of Nanostructured Materials, Faculty of Physics, University of Vienna, Austria

$Fe_{73.4}Cu_1Nb_3Si_{15.5}B_{6.6}$ is a widely used industrial soft magnetic material with high saturation polarization (in the nanocrystalline state). The starting material is an amorphous ribbon produced by melt spinning. Magnetic properties are commonly controlled by annealing (which leads to a nanocrystalline state) under an external magnetic field[1]. Applying severe plastic deformation can be used to influence and induce the crystallisation process in the amorphous alloy. However due to its high value of elastic constants this material has been a challenge for mechanical deformation process up to now. It is the scope of this work to study experiments for creating bulk sample of – possibly partly crystallised - $Fe_{73.4}Cu_1Nb_3Si_{15.5}B_{6.6}$ in order to investigate the change of crystallization process and magnetic properties under mechanic deformation.Samples were created by subjecting stacks of tapes of amorphous $Fe_{73.4}Cu_1Nb_3Si_{15.5}B_{6.6}$ to high pressure torsion (HPT) under different loading conditions and temperatures.

DSC investigations were performed to optimize the deformation temperature. All samples were analysed for cracks as well as investigated by XRD searching for an induced crystallisation process. The undeformed precursor was investigated in synchrotron diffraction experiments at DESY to ensure the absence of any nano-sized precipitates. These ex-situ XRD investigations of the HPT deformation experiments indicate a significant change of the microstructure. The experimental results are discussed combining the effect of severe plastic deformation with the influence on the microstructure. An in-situ annealing experiment of HPT deformed samples is proposed to further improve the understanding of the crystallization dynamics in these type of alloys.

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Surface Crystallization Studies of 2-decyl-7-phenyl-[1]benzothieno[3,2-b][1]benzothiophene (Ph-BTBT-10)

W. R. Bodlos,[1] A. Hofer, [1] L. Beverina,[2] M. Ramsey,[3] R. Resel,[1]
[1] *Institut für Festkörperphysik, Technische Universität Graz, Austria*
[2] *Department of Materials Science, Università di Milano-Bicocca, Italy*
[3] *Institute of Physics, Karl-Franzens-University Graz, Austria*
bodlos@student.tugraz.at

Benzothieno[3,2-b][1]benzothiophene (BTBT) derivatives are very promising candidates in the field of solution processable organic semiconductors. [1] In this work the surface crystallization behaviour of Ph-BTBT-10 is investigated. Thin films starting from the monolayer regime up to thick films were prepared by spin coating and physical vapour deposition. The films were investigated in terms of crystalline properties and thin film morphology by X-ray diffraction, X-ray reflectivity and Grazing Incidence X-ray diffraction, atomic force microscopy and optical microscopy. It is shown that by spin coating at low concentrations of 0.3 g/l an incomplete monolayer of standing molecules with the aromatic core pointing towards the substrate is formed. At higher concentrations between 0.5 g/l and 3 g/l strong island growth starts which leads to the formation of macroscopic islands with dendritic monolayers in-between them. Physical vapour deposition demonstrates a more homogenous distribution of the material forming terraces of multiple molecules. In thin films whether in solution or PVD crystalline structures are observed in XRD. As well as the known bulk phase with a head to head arrangement of two molecules in an orthorhombic unit cell possessing a herringbone structure, also other polymorphic phases are present.

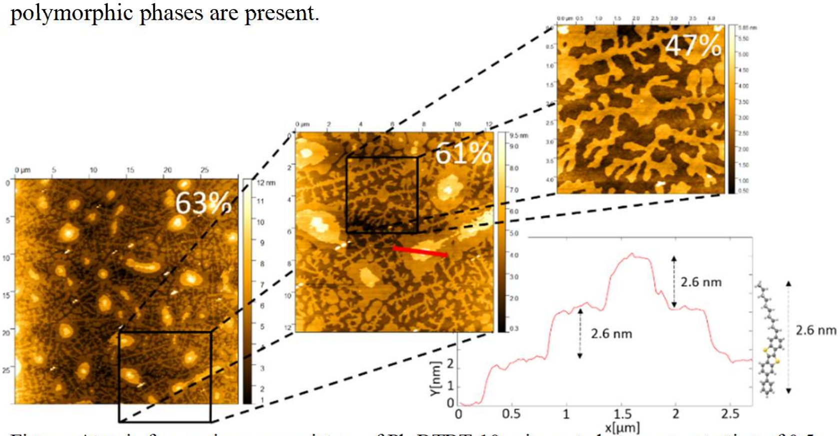

Figure: Atomic force microscope picture of Ph-BTBT-10 spin coated at a concentration of 0.5 g/l onto a SiO_2 substrate. The dendritic structure with islands growing overhead reaches a coverage of 63%.

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Synthesis and Characterization of Crystalline Shape-Anisometric Polymer Particles as Model Systems for Self-Assembled Materials

Tobia Cavalli[1,2] and Miriam M. Unterlass[1,2,3*]
[1] *Institute of Materials Chemistry, Technische Universität Wien, Austria*
[2] *Institute of Applied Synthetic Chemistry, Technische Universität Wien, Austria*
[3]*Center for Molecular Medicine of the Austrian Academy of Sciences (CeMM), Lazarettgasse 14, AKH*

*miriam.unterlass@tuwien.ac.at

This project deals with the synthesis of shape-anisotropic polymer particles as model systems for self-assembly. To achieve this goal, we profit from the fact that crystalline compounds are of non-spherical shapes. The preparation of arrays of anisotropic crystalline particles in the colloidal domain (1 nm to 1 μm) proceeds by first synthesizing so-called monomer salt (MS) crystals. MS crystals present a set of peculiar characteristics, such as (*i*) higher stability and storability in comparison to classic polymer precursors; (*ii*) proximity of reactive functional groups, and (*iii*) ideal stoichiometry. MS crystalline particles are first obtained by precipitation upon mixing of two aqueous co-monomer solutions, *i.e.* an acid (pyromellitic acid, PMA) with a base (*p*-phenylene diamine, PDA). This step is followed by solid-state polymerization (SSP), which bears the crucial characteristic of yielding polymer particles that retain the shape of the initial MS crystals. SSP is a solvent-free technique recently introduced by our group that involves temperature treatment of the MS particles below their melting point.[1] Therefore, by aiming at the control of the habit of MS crystals, one is able generate a plethora of shape-anisotropic particles. To modify the shape of the crystalline MS particles, we employ additives as crystal-habit modifiers during the crystal growth process.[2] The use of additives is a well-known technique in the crystal growth field. By preferentially attaching additives to different facets of the growing crystal, *i.e.* to facets with different surface-free energies G_i, additives allow for the modification of crystal habit by inhibiting the growth in specific directions.

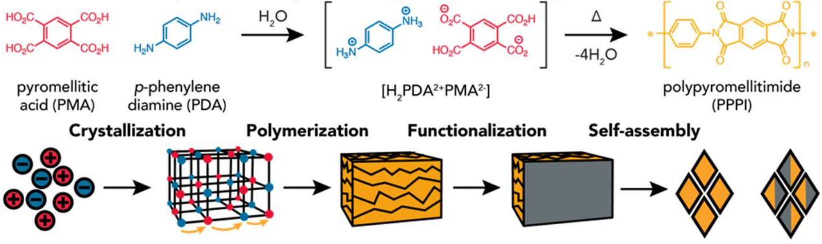

Figure 2. A. Acid-base reaction in aqueous solution between pyromellitic acid (PMA) and *p*-phenylene diamine (PDA) yields the monomer salt [$H_2PMA^{2+}PDA^{2-}$], which is then polymerized *via* solid-state to poly(pyromellitimide) (PPPI). B. After formation of a monomer salt (MS) crystalline particles, these are polymerized in the solid-state with shape-retention . Further steps towards increasing the anisotropy of the particles include functionalization of specific surfaces, generating sets of shape- and function-anisotropic particles which will then lead to self-assembled phases.

For the goal of generating self-assembled structures, control over the crystalline structure and size range of the polymeric particles is a desired feature. Crystalline structure determines the type of interactions between exposed facets of different particles. Preliminary results show that MS particles already self-assemble into ordered phases upon nothing but drying – driven, as we believe through simply their anisotropic shapes thriving to pack closely. The size range of the particles also plays an important role. To date, MS particles have been limited to sizes in the ranges of 200-300 µm, a scale far out the colloidal range required for self-assembly.

With this contribution, we present a study on the effect of additives on the crystal growth of [$H_2PMA^{2+}PDA^{2-}$]. We investigated the effect of different parameters on the shape and size of the final particles, such as co-monomers and additive concentrations. All particles have been characterized by optical microscopy, scanning electron microscopy, wide angle X-ray diffraction and infrared spectroscopy. Our results show that crystal growth of the prototypical [$H_2PMA^{2+}PDA^{2-}$] system is indeed affected by specific additives. Overall, we aim at the generation of large sets of anisotropic particles with same chemical composition, that will further serve as building blocks towards the generation of novel materials by self-assembly.

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Zernike Phase Contrast Nanotomography of low absorbing biological functional materials

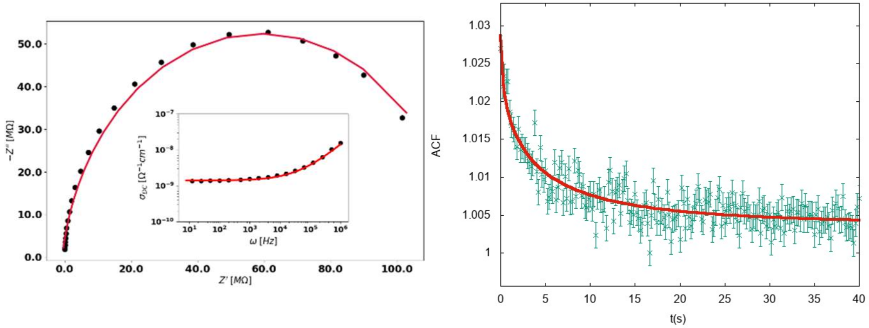
Silja Flenner

[1] *Institut für Werkstoffforschung, Helmholtz-Zentrum Geesthacht, Germany*
silja.flenner@hzg.de

Measuring Ionic Conductivity in Rubidium Borate Glasses via IS and aXPCS

Tobias Fritz*[1]*, Katharina Holzweber[1], Bogdan Sepiol[1], Christoph Tietz[1]
[1] Faculty of Physics, University of Vienna, Boltzmanngasse 5, 1090 Vienna
a01217228@unet.univie.ac.at

Measuring the complex impedance $Z^*(\omega)$ of alkali doped rubidium borate glasses via IS (Impedance Spectroscopy) within a wide frequency range (0.1Hz – 1MHz) enables determining of the ionic conductivity σ_{DC}. From the Arrhenius behaviour activation energy E_A and the diffusion coefficient D can be calculated[1]. Further analysis of the data can be done using the dielectric modulus formalism, which relates dielectrically relaxations in the time and in frequency domains. Another novel and efficient technique for measuring dynamical properties in matter is aXPCS (atomic-scale X-ray Photon Correlation Spectroscopy) enabled by coherent synchrotron radiation sources[2]. Speckle pattern is correlated and give information about the atomic motion inside the material being, however, influenced by the synchrotron beam.



<u>Figure 1:</u> Left: Impedance Spectrum of 15Rb2O85B2O3 @643K. The inset depicts the respective conductivity spectrum as a function of angular frequency ω. In the limit of low frequencies the conductivity becomes constant. **Right:** Autocorrelation function obtained from aXPCS measurement. The fitted line denotes the decay according to Kohlrausch-Williams-Watt law.

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Determination of Chromium and Zinc Speciation in Airborne Particulate Matter by X-Ray Absorption Near-Edge Structure

G. Gunchin*, [1], J. Osan[2], A. Migliori[3], D. Shagjjamba[4], and C. Streli[5]

[1] Vienna University of Technology, Vienna, Austria
[2] Environmental Physics Department, Hungarian Academy of Sciences Centre for Energy Research, Budapest, Hungary
[3] Nuclear Science and Instrumentation Laboratory, Physics section, IAEA, Seibersdorf, Austria
[4] Nuclear Research Center, National University of Mongolia, Ulaanbaatar, Mongolia
[5]Vienna University of Technology, Atominstitut, Vienna, Austria

**Corresponding author:* g.gerelmaa@gmail.com

Abstract

This study presents the first time the result of elemental speciation in atmospheric particulate matter (APM or PM) samples from Ulaanbaatar, Mongolia. Samples of $PM_{2.5}$ (fine) and $PM_{10-2.5}$ (coarse) were examined by X-ray Fluorescence analysis using PAnalytical Espilon 5 spectrometer to investigate concentrations of chromium (Cr) and zinc (Zn) in the atmosphere. X-ray Absorption Near-edge Structure (XANES) spectrometry was used to obtain speciation information of elements. Analyses of recorded data using linear combination fitting method were applied to identify potential compounds in samples The preliminary XANES results show that major chemical forms of Cr in the both fine and coarse fractions are chromium(III) sulfate ($Cr_2(SO_4)_3$) and chromium(III) oxide (Cr_2O_3). For chemical forms of Zn, the contribution of Zn sulfate ($ZnSO_4$) was dominating to the fine fractions, whereas Zn franklinite ($ZnFe_2O_4$) found to be dominating compounds to the coarse fractions.

Keywords: XANES, Cr and Zn speciation, $PM_{2.5}$, $PM_{10-2.5}$, Ulaanbaatar

Phase Transition Behavior of an Asymmetric Benzothienobenzothiophene (BTBT) Derivative

S. Hofer[1], W. Bodlos[1], L. Beverina[2], and R. Resel[1]
[1]Institute of Solid State Physics, Graz University of Technology, Austria
[2]Department of Materials Science, Università di Milano-Bicocca, Italy
sebastian.hofer@tugraz.at

The molecule Ph-BTBT-C10 (2-decyl-7-phenyl-[1]benzothieno[3,2-b][1]benzothiophene) is an asymmetric molecule, which shows a fully reversible soft crystalline phase transition from the bulk phase, which results in a thin film phase with high thermal stability as well as large carrier mobility in thin film transistors[1]. Thin-film samples are prepared by spin coating at different spin speeds and solution concentrations. The phase transition behavior as well as the crystal structure of the Smectic E (SmE) phase is studied in relation to the bulk structure, by in-situ Grazing Incidence X-ray Diffraction experiments, which were carried out using synchrotron radiation at different temperatures. Additionally, in-situ specular X-ray diffraction patterns are measured on in-house equipment to characterize the crystal structures as a function of temperature. While the phase transition can be monitored precisely by an observed peak shift, the exact molecular packing in the SmE cell is still not fully solved. Starting from Molecular Dynamic (MD) simulations[2] potential unit cells for the SmE phase are compared to experimental results. Distinct Bragg peaks in the diffraction pattern indicate an ordered structure in the out-of-plane as well as the in-plane direction of the sample, with very similar in-plane order as in the case of the bulk phase, but significant differences in out-of-plane order. Specific spin conditions lead to a disordered phase with broad diffraction peaks. This disordered phase cannot be transferred to an ordered state by temperature treatment.

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In vitro degradation experiments over one month period on magnesium-based alloys for biodegradable implant applications

D. Krüger[1], B. Zeller-Plummhoff[1], J. Moosmann[1], D.C.F. Wieland[1], J. U. Hammel[3], R. Willumeit-Römer[1]

[1]Institut für metallische Biomaterialien, Helmholtz-Zentrum Geesthacht, Hamburg, Germany
[2]Malmö University, Malmö, Sweden
[3]Institut für Werkstoffphysik, Helmholtz-Zentrum Geesthacht, Hamburg, Germany

Magnesium (Mg)-based alloys can be applied as promising biodegradable implants due to their good biocompatibility, a Young's modulus similar to the one of bone and the ability for complete degradation [1]. The major advantage is the prevention of a second and costly removal surgery. This kind of application of Mg in medicine is only viable if the implant degradation takes place in a controlled and sufficiently slow manner [1]. To examine the degradation behaviour of two Mg-gadolinium (Gd) alloys with varying Gd content (5, 10 wt% Gd content) *in vitro* semi-static degradation tests over one month period were held. The samples had a shape of a screw (2x4 mm) and as degradation medium mimicking biological conditions α-MEM (Minimum Essential Medium) + 10% FBS (Fetal Bovine Serum) + 1% Pen Strep (Penicilin Streptomycin) was used. Investigations were performed using high-resolution synchrotron radiation computed tomography at beamline P05 at PETRA3 Deutsche Elektronen-Synchrotron (DESY), Hamburg. The analysis on the experimental data aim to extract the DR (degradation rate), DH (degradation homogeneity), dependency of Gd agglomerations on DR and DH. Also microstructural investigations were carried out using optical microscopy (OM), scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). First results indicate that Mg-5Gd exhibits bigger grain size (GS) (52.6±15.3 μm) than Mg-10Gd (GS = 25.9±0.1 μm). DH analysis show that Mg-5Gd screws tend to degrade with localized pitting corrosion in the first week of immersion. Afterwards the corrosion layer starts to spread over the whole surface. In contrast, the primarily localized pitting corrosion depth of Mg-10Gd is smaller, having a larger surface distribution (Fig. 1). An average DR = 0.30±0.09 mm/year for both alloys is observed over all 4 time points during one month period, which is acceptable for biodegradable implants [2].

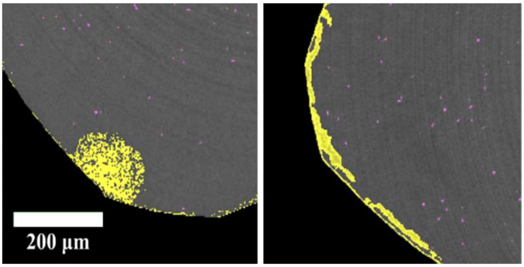

Fig. 1: Exemplary cross sectional slices of screws after 1 week degradation: left) Mg-5Gd; right) Mg-10Gd. Yellow colored areas represent corrosion layers; magenta colored points represent Gd agglomerations.

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A New Ferromagnetic Insulator with Low Damping: Growth, Magnetic and Structural Properties of Zn/Al Doped Nickel Ferrite

J. Lumetzberger[1], M.Buchner[1], V.Ney[1] and A. Ney[1]

[1]*Institute of Semiconductor and Solid State Physics, Johannes Kepler University Linz, 4040 Linz, Austria*

julia.lumetzberger@jku.at

In spintronics one aims to obtain pure spin currents as additional degree of freedom in logic circuits aside from electric charges. By means of spin pumping it is feasible to induce a spin current from ferromagnetic materials into adjacent non-magnetic layers. To ensure pure spin currents and exclude charge currents ferromagnetic insulators are the material of choice. However ferromagnetic insulators with low intrinsic damping are sparse. The most commonly used material for magnetoelectric devices is yttrium iron garnet (YIG)[1]. YIG has two major drawbacks, namely the complex garnet structure, which is typically grown on Gadolinium Gallium Garnet (GGG) substrates and its weak magnetoelastic response. It could be replaced by cubic NiZnAl – ferrite thin films grown on $MgAl_2O_4$, which were reported to have similar magnetic properties[2]: ferromagnetic at room temperature, low intrinsic damping, and additionally a strong magnetoelastic coupling. The combination of these properties makes it especially interesting for application as high frequency devices and in magnetoelectric and acoustic spintronics. Since the NiZnAl-ferrite has also insulating properties it can be used as source for pure spin currents in devices. In this contribution we use two preparation methods, reactive magnetron sputtering and pulsed laser deposition, to optimize the magnetic properties by the variation of growth parameters like oxygen ratio, temperature and annealing time. All samples are analyzed with x-ray diffractometry for their crystallographic properties and SQUID magnetometry for ferromagnetism. Additionally transmission electron microscopy is performed to investigate the interface on an atomic scale. To evidence the desired low magnetic damping room temperature multifrequency ferromagnetic resonance (FMR) with a short-circuited set-up is performed as well[3]. From the frequency dependence of the resonance line-width the homogeneous and inhomogeneous contributions to the damping of the system can be extracted. Furthermore the angular and frequency dependence of the resonance position is measured to quantify the strain-induced magnetic anisotropy contributions.

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Structural Differences of Human and Yak Hair

Alexander R.M. Müllner[1],Stephan Puchegger[2], Herwig Peterlik[1]

[1] DCS, Faculty for Physics, University of Vienna, Austria
[2] Faculty Center for Nano Structure Research, University of Vienna, Austria
alexander.muellner@univie.ac.at

Yak belly hair has been proposed to replace human hair to study the effects of chemicals and hair dyes[1]. The particular advantage of Yak belly hair is the absence of the coloring molecule melanin, which is unfavorable for example for investigations using Raman spectroscopy. To replace human hair, one has to ensure that the properties of Yak hair closely resemble those of human hair. This was the starting point for our investigations: We used various techniques such as small-angle X-ray scatting (SAXS) and scanning electron microscopy (SEM) to get information on the structural differences. It turned out that Yak hair is in general much more porous than human hair, which has to be taken into account in dyeing experiments. Furthermore, we performed tensile tests of both types of hair to determine their mechanical properties. The results for Yak and human hair are thoroughly compared, in particular with respect to their hierarchical structure, which is typical for many biological tissues.

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Residual stress fields of single steel ball impacts in Al alloys

Marc-André Nielsen

[1] *Institut für Werkstoffforschung, Helmholtz-Zentrum Geesthacht, Germany*
marc-andre.nielsen@hzg.de

In situ experiment for selective laser melting

Jan Rosigkeit

[1] *Institut für Werkstoffforschung, Helmholtz-Zentrum Geesthacht, Germany*
marc-andre.nielsen@hzg.de

The Software Package GIDVis and its Application to Rotating Grazing Incidence X-Ray Diffraction Data

Benedikt Schrode[1], *Stefan Pachmajer[1], Michael Dohr[1], Christian Röthel[2], Jari Domke[3], Torsten Fritz[3], Roland Resel[1], Oliver Werzer[2]*
[1] Institute of Solid State Physics, Graz University of Technology, Austria
[2] Institute of Pharmaceutical Sciences, Department of Pharmaceutical Technology, University Graz, Austria
[3] Institute of Solid State Physics, Friedrich Schiller University Jena, Germany
b.schrode@tugraz.at

Grazing incidence X-ray diffraction (GIXD) is an experimental method frequently applied to characterize a wide variety of thin films and surfaces. Depending on the texture of the sample's crystallites, special care is required during the experimental measurements and successive data evaluation. For example, samples with large individual crystallites on a surface resulting in poor statistics or epitaxially grown samples with defined in-plane alignment of the crystallites require both a full sample rotation around the surface normal during data collection. This is necessary to obtain all information for phase, texture and epitaxy analysis. Moreover, this experimental approach opens new possibilities for advanced characterisation methods which in simple static experiments are inaccessible, e.g. the determination of the in-plane mosaicity. GIDVis is a software package developed in MATLAB for visualization and analysis of GIXD data in general but mostly used for thin films. It is specialized on data obtained during a sample rotation around the surface normal, i.e. a rotating GIXD experiment. Due to the geometrical independent character of the underlying algorithms implemented, most features can be used independently from the input data type. GIDVis allows the user to perform detector calibration, data stitching, intensity corrections, standard data evaluation (e.g. cuts and integrations along specific reciprocal space directions), crystal phase analysis, etc. Further, these features are not limited to a single experimental setup, data from mostly all diffraction setups can be processed. To fully take advantage of the measured data in the case of sample rotation, pole figures can be calculated from the experimental data for any q value covered. As an example, GIDVis is used here to investigate the epitaxial alignment of pentacenequinone (P2O) crystallites on a Au(111) surface using pole figures (cf. Figure 1), illustrating the importance of the sample rotation. GIDVis is available online free of charge (http://www.if.tugraz.at/amd/GIDVis/).

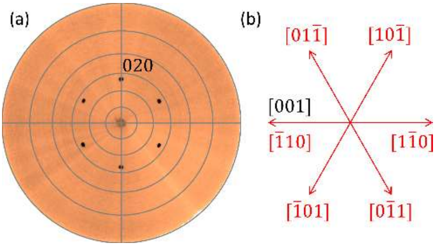

Figure 1: (a) Pole figure of the 020 peak of pentacenequinone (P2O) on Au(111) and

(b) derived alignment of the P2O unit cell (black) on the gold surface (red).

Indexing Grazing Incidence X-ray Diffraction Patterns of Thin Films: Lattices of Higher Symmetry

Josef Simbrunner[1], Sebastian Hofer[2], Benedikt Schrode[2], Roland Resel[2], Ingo Salzmann[3]
[1] *Department of Neuroradiology, Vascular and Interventional Radiology, Medical University Graz, Austria*
[2] *Institute of Solid State Physics, Technical University Graz, Austria*
[3]*Department of Physics, Department of Chemistry & Biochemistry, Centre for NanoScience Research (CeNSR), Concordia University Montreal, Canada*
josef.simbrunner@medunigraz.at

Grazing incidence X-ray diffraction studies on organic thin films are often performed on systems showing fibre-textured growth. However, indexing their experimental diffraction patterns is generally challenging, especially if low-symmetry lattices are involved. Recently, analytical mathematical expressions for indexing experimental diffraction patterns of triclinic lattices were provided[1]. In the present work, the corresponding formalism for crystal lattices of higher symmetry is given and procedures for applying these equations for indexing experimental data are described[2]. Two examples are presented to demonstrate the feasibility of our indexing method. For layered crystals of the prototypical organic semiconductors diindenoperylene and (*ortho*-difluoro)sexiphenyl, as grown on highly oriented pyrolytic graphite, their yet unknown unit-cell parameters are determined and their crystallographic lattices are identified as monoclinic and orthorhombic, respectively.

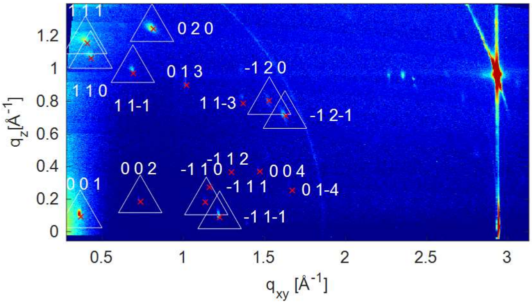

Figure. X-ray diffraction of diindenoperylene crystals (DIP) grown on a (001) substrate of highly oriented pyrolytic graphite (HOPG. Reciprocal space map measured by grazing incidence X-ray diffraction with a selection of peaks which were used for indexing. The centres of the triangles and the crosses give the experimental and calculated peak positions, respectively; the respective Laue indices are given additionally.

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3D X-ray Diffraction Microscopy (3DXRD) Using High Resolution X-ray Nanodiffraction

Hergen Stieglitz

[1] *Institut für Werkstoffforschung, Helmholtz-Zentrum Geesthacht, Germany*
hergen.stieglitz@hzg.de

Anton Paar

BRUKER