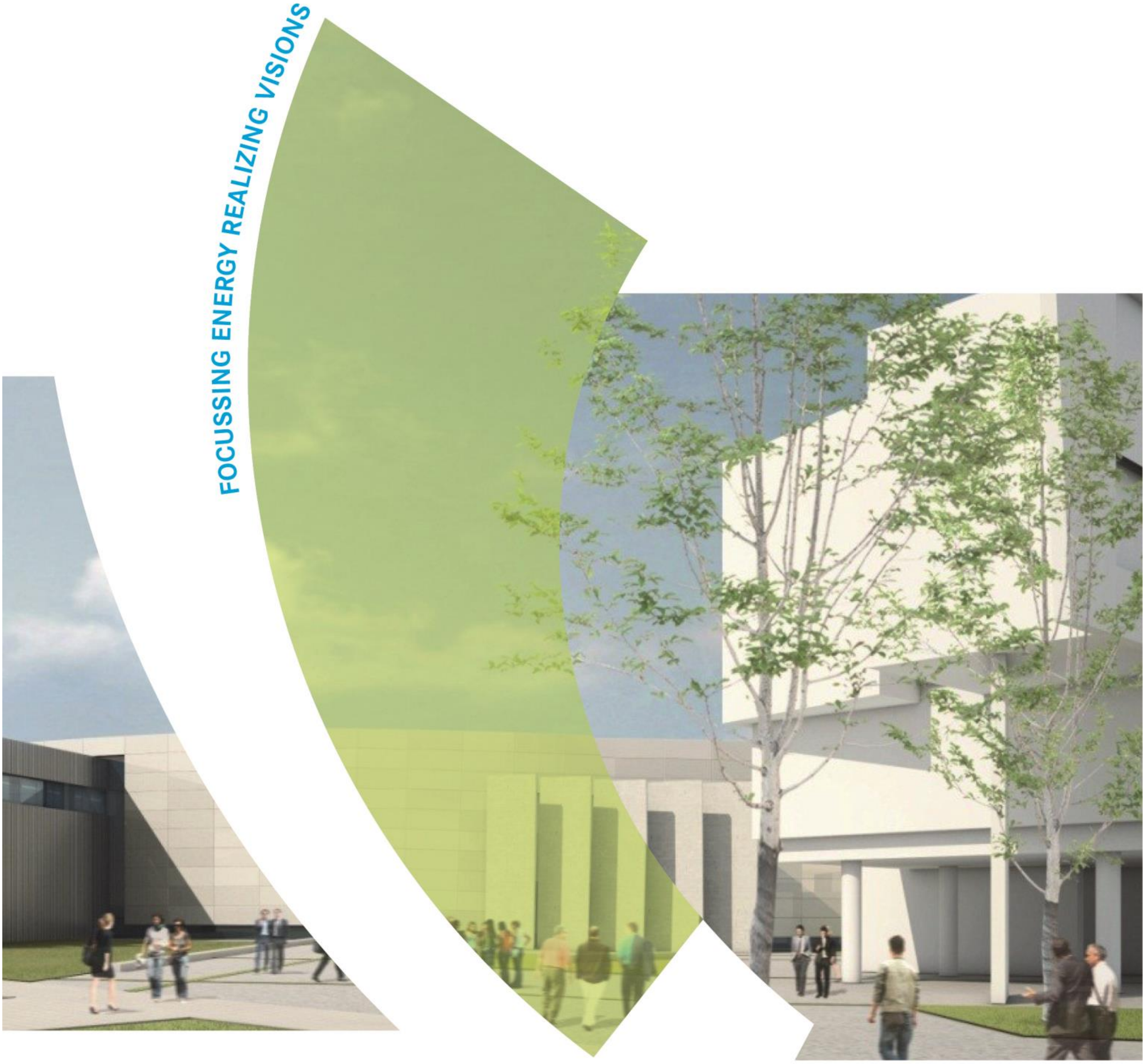
HZB Helmholtz Zentrum Berlin



IMAGING WORKSHOP

BESSY II

October 5 - 6, 2015
Berlin-Adlershof

Dear participant,

HZB kindly welcomes you to our foresight workshop “IMAGING at BESSY II”.

With nanoscale resolution, modern spectromicroscopies based on photon-in/photon-out and photon-in/electron-out techniques deliver unique elemental, chemical, structural, magnetic, and electronic information.

The workshop “IMAGING at BESSY II” focusses on recent advances and future prospects in synchrotron imaging techniques and their applications. World leading experts will illustrate current achievements and future developments in photoemission electron microscopy, X-ray microscopy, coherent X-ray imaging methods (ptychography, holography) and synchrotron X-ray tomography. The workshop provides the opportunity to exchange ideas on the interplay between scientific requirements and the need for novel instrumentation to open up new opportunities for imaging, especially at future coherent light sources.

We invite you to explore these opportunities with us at the workshop.

The Workshop “IMAGING at BESSY II” is the third in a series of foresight workshops at BESSY II which are designed to establish a continuous discussion platform for future projects and research activities in concert with current and future users from universities, research institutes, and industry. The aim of the dialogue is to identify future scientific fields as well as expectations, needs, and requirements for cutting edge science with synchrotron radiation.

We hope that this workshop will stimulate your interest in future developments at BESSY II and initiate fruitful discussions and new experiments and collaborations. Thank you all for joining us and enjoy the meeting.

Sincerely,

Prof. Dr.-Ing. Anke Kaysser-Pyzalla
Scientific Director and Chief Executive

Content

Imaging Workshop 5/6.10.2015

Programme

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Holography	Stefan Eisebitt	TU Berlin	9:10-9:40
PEEM	Ernst Bauer	Arizona State University	9:40-10:10
TXM	Eva Pereiro	ALBA	10:10-10:40
Coffee			10:40-11:00
Ptychography	Chris Jacobson	Argonne Lab/Northwestern U	11:00-11:30
µm Tomography/ Multiprobe Scanning Imaging	Peter Fratzl	MPI Golm	11:30-12:00
Imaging at Future Light Sources	Christoph Quitmann	MAXlab	12:00-12:30
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Ideas, Suggestions and Requirements			15:00-15:15
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PEEM (Chair: Ernst Bauer)	Claus Schneider Armin Kleibert Ruud Tromp Richard Harrison	FZJ PSI IBM/Leiden University University of Cambridge	15:45-17:45
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Programme

Topic	Speaker	Affiliation	Time
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Ideas, Suggestions and Requirements			12:45-13:00
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µm Tomography/Multiprobe Scanning Imaging (Chair: Peter Fratzl)	Martin de Jonge Ingo Manke Wolfgang Ludwig	Australian Synchrotron HZB ESRF	14:00-15:30
Ideas, Suggestions and Requirements			15:30-15:45
Coffee			15:45-16:15
Into the Future			
BESSY II Photon Science Instrumentation and Optics	Frank Siewert	HZB	16:15-16:45
BESSY II Accelerator and Storage Ring Instrumentation	Andreas Jankowiak	HZB	16:45-17:15
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Abstracts of the overview talks

Monday, 5th of October

Chair: Simone Raoux, Bernd Müller

Location: BESSY II Lecture hall

X-Ray Holography

Stefan Eisebitt[1,2,3]

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The *coherent* photon flux that can be extracted from a chaotic light source is proportional to the source brightness (or spectral brilliance) times the wavelength squared.[1] With ring-based synchrotron radiation sources becoming brighter with every generation towards the diffraction limit (at a given wavelength) and with free electron x-ray lasers in operation, interference experiments with x-rays have become increasingly feasible. Holography uses the interference of an object with a reference wave for direct, non-iterative image formation, allowing to extract amplitude and phase of the wave field. Several different schemes exist and are now routinely used for research beyond the demonstration of the techniques themselves.[2] I will explain the basics of and give an overview on the different x-ray holography approaches, with some emphasis on Fourier Transform Holography and time resolved imaging at synchrotrons and FELs.[3,4] In an outlook, I will try to discuss some future trends.

References:

[1] B. Lengeler, Naturwissenschaften **88**, 249 (2001)
[2] B. Pfau, S. Eisebitt, X-Ray Holography. In: Synchrotron Light Sources and Free-Electron lasers, 1st edn. (Springer, Berlin, 2015) doi: 10.1007/ 978-3-319-14394-1_18-1 (in print)
[3] F. Büttner et al., Nature Physics **11**, 225 (2015)
[4] C. von Korff Schmising et al., PRL **112**, 217203 (2014)

PEEM

Ernst Bauer

Arizona State University, USA

PEEM at third generation synchrotron radiation sources is discussed from the point of view of a user interested in surface and thin film phenomena. Emphasis is put on the combination with LEEM and spectroscopy (SPELEEM) and reciprocal space imaging (k-space imaging), which allows not only electronic, chemical and magnetic but also structural characterization. The present limitations of the spatial, energy and time resolution as determined by lens aberrations, space charge and image detection system, energy filter, photon and electron beam properties and other instrument parameters, will be presented. Possibilities for overcoming some of these limitations with further instrument development will be indicated.

General reference:

Ernst Bauer, Surface Microscopy with Low Energy Electrons, Springer, New York, 2014

Soft X-ray microscopy, an imaging tool for biology and magnetism

A.J. Pérez-Berná[1], A. Sorrentino[1], M.J. Rodríguez[2], F.J. Chichón[2], J.L. Carrascosa[2], P. Gastaminza[2], J. Otón[2], J.M. Carazo[2], C. Blanco-Roldán[3], M. Vélez[3], C. Quirós[3], S. Ferrer[1], E. Pereiro[1]

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3 Departamento de Física, Universidad de Oviedo, 33007 Oviedo, Spain

Transmission soft X-ray microscopy is versatile imaging tool that can provide valuable information in different scientic areas as, for instance, structural cell biology and magnetism as illustrated with the two examples below.

Cryo soft X-ray tomography (cryo-SXT) [1] has proven to shed light on complex phenomena such as microbial infections [2, 3]. In the case of Hepatitis C virus (HCV), we have obtained recently the first complete cartography of the dramatic cellular modifications caused by the stable subgenomic HCV replicon transfected in cell culture at different steps of the viral life cycle [4]. In addition, we have also investigated the recovery at cellular level of HCV replicating cells treated with specific antiviral drugs. Structural studies of viral factories in whole cells provide a powerful tool for the analysis of host-pathogen interactions, allowing for a potential platform for the trial of new antiviral drugs and vaccines.

Advances in nanoscale magnetism increasingly require characterization tools providing detailed descriptions of magnetic configurations. Magnetic transmission X-ray microscopy produces element specific magnetic domain images with nanometric lateral resolution in films of hundred nanometers. Using the angular dependence of magnetic contrast in a series of high resolution tilt projections, we have obtained the canting angles and sense of the magnetization and its dependency with the films thickness [5]. This method has allowed identifying complex topological defects (merons or ½ skyrmions) in a $NdCo_5$ film that are only partially replicated by the Permalloy overlayer. These results open new characterization possibilities of deeply buried magnetic topological defects, nanostructures and devices.

References:

[1] Schneider G. *et al*. Nature Methods **7**, 985-987 (2010)
[2] Chichón F.J. *et al*. J. Struct. Biol. **177**, 202-211 (2012)
[3] Cruz-Adalia A. *et al*. Cell Host & Microbe **15**, 611-622 (2014)
[4] Pérez-Berná A.J. *et al.* (submitted)
[5] Blanco-Roldán C. *et al*., Nature Comm. DOI: 10.1038/NCOMMS9196 (2015)

A more complete picture: combining ptychography and fluorescence on frozen hydrated specimens at the Advanced Photon Source

Junjing Deng[1], David J Vine[2], Si Chen[2], Youssef S G Nashed[3], Qiaoling Jin[1], Tom Peterka[3], Rob Ross[3], Stefan Vogt[2], and Chris J Jacobsen[2,1]

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2 X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, USA
3 Mathematics and Computer Science Division, Argonne National Laboratory, USA
4 Chemistry of Life Processes Institute, Northwestern University, Evanston Illinois, USA

X-ray fluorescence microscopy reveals trace elements with no dependence on binding affinities (unlike with visible light fluorophores) and with improved sensitivity relative to electron probes. However, X-ray fluorescence is not very sensitive for showing the light elements that comprise the majority of cellular material. X-ray ptychography can be combined with fluorescence to image both cellular structure and trace element distribution in frozen-hydrated cells at cryogenic temperatures [1], with high structural and chemical fidelity. Ptychographic reconstruction codes [2] and fast scanning approaches [3] deliver phase and absorption contrast images at a resolution beyond that of the illuminating lens or beam size. Using 5.2-keV X-rays, we have obtained sub-30-nm resolution structural images and ∼90-nm-resolution fluorescence images of several elements in frozen-hydrated green algae. This combined approach offers a way to study the role of trace elements in their structural context, and will become even more powerful with the development of diffraction-limited storage rings [4].

References:

[1] PNAS 112, 2314 (2015).
[2] Optics Express 22, 32082 (2014)
[3] Optics Express 23, 5438 (2015)
[4] J. Sync. Rad. 21, 1031 (2014)

Complex (bio)materials – Perspectives for synchrotron-based multiprobe imaging

Peter Fratzl

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

The permanent adaptation of tissues in animals or plants, including in our own body creates materials with an extreme heterogeneity in the three space dimensions and in time. In order to improve our understanding of biological materials such as bone, tendon or other organs as well as their diseases, biological approaches need to be complemented by imaging techniques that provide multi-scale and multimodal information about structure, composition and physical properties. Such imaging methods are also needed for the study of many complex engineering materials and material systems. While synchrotron radiation is not generally suited for in-vivo imaging, it provides an exceptionally flexible tool to image composition, structure and properties of millimeter size specimens in several dimensions. Such approaches include various types of micro- and nano-tomography, spectromicroscopy and scanning imaging techniques. The talk will showcase several examples, including bone from our skeleton, where scanning-diffraction and spectroscopy, in combination with tomography improves our understanding of the material and the effect of disease and treatment. Further development of these synchrotron-based methodologies is under way and this will provide a unique opportunity for research in biomaterials and in complex material systems in general.

MAX IV – Better light for better microscopy

C. Quitmann et al.

MAX IV Laboratory, Lund University, Lund, Sweden

X-Ray microscopy is a powerful technique, which allows pushing the boundaries of our knowledge about nature and its materials. However it puts high demands on the x-ray source.

Recent developments in storage rings are pushing their performance to achieve higher brightness and coherence in ever-harder x-ray regions [1]. I will present the recent progress in electron storage rings as exemplified by the MAX IV project [2] currently under commissioning [3]. In addition I will present potential future applications of x-ray microscopy based on such rings.

References:

[1] Eriksson, M., van der Veen, J. F. and Quitmann, C. (2014), Diffraction-limited storage rings – a window to the science of tomorrow. Jnl of Synchrotron Radiation, 21: 837–842. doi: 10.1107/S1600577514019286, and other contributions in this special volume.
[2] Tavares, P. F., Leemann, S. C., Sjöström, M. and Andersson, Å. (2014), The MAX IV storage ring project. Jnl of Synchrotron Radiation, 21: 862–877. doi: 10.1107/S1600577514011503 Nano Lett, 8, 11, (2008)
[3] Nature, Breaking news 2015-08-26

Abstracts of the Holography and Coherent Diffraction Imaging Session

Monday, 5th of October

Chair: Stefan Eisebitt

Location: BESSY II Lecture hall

X-ray imaging at the nanoscale: ptychography, holography and tomography

Tim Salditt

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X-rays deeply penetrate matter and thus provide information about the functional (interior) architecture of complex samples, from biological tissues and cells to novel composite materials. However, this potential of hard x-rays in view of penetration power, high spatial resolution, quantitative contrast, and compatibility with environmental conditions has to date not been fully developed, mainly due to significant challenges in x-ray optics. With the advent of highly brilliant radiation, coherent focusing, and lens-less diffractive imaging this situation has changed. We show how nano-focused coherent x-ray synchrotron beams can be used for scanning as well as for full field holographic x-ray imaging. The central challenge of inverting the coherent diffraction pattern is discussed. Different reconstruction algorithms are presented, from holographic techniques to ptchography, a phase retrieval approach adapted from electron microscopy, which has proven extremely useful in x-ray imaging [1].

Following an introduction to the basic concepts of lensless x-ray imaging, different recent examples of biological imaging are presented, ranging from bacterial [1,2] and eukaryotic cells [3], to the level of tissue and organs [4].

In particular, we show how holographic projection images recorded by using the quasi-point source of an x-ray waveguide can be inverted to quantitative two and three dimensional images of the object, see Fig.1. The experimental and conceptual aspects of image formation, object reconstruction, contrast transfer function and resolution will be discussed, and illustrated by different examples.

References:

[1] K. Giewekemeyer,et al. Quantitative biological imaging by ptychographic x-ray diffraction microscopy,
PNAS 107 (2), 529 (2010)
[2] R. N. Wilke, M. Priebe,M. Bartels,K. Giewekemeyer, A. Diaz, P. Karvinen, T. Salditt
Hard X-ray imaging of bacterial cells: nano-diffraction and ptychographic reconstruction
Optics Express 20, 19232-19254 (2012)
[3] M. Bartels, M. Krenkel, J. Haber, R. N. Wilke, and T. Salditt
X-Ray Holographic Imaging of Hydrated Biological Cells in Solution
Phys. Rev. Lett. (2015), 114, 048103
[4] M. Krenkel, A. Markus, M. Bartels, C. Dullin, F. Alves, T. Salditt
Phase-contrast zoom tomography reveals precise locations of macrophages in mouse lungs
Sci. Rep. (2015), 5, 09973

Holographic X-ray imaging of magnetic nanostructures

Bastian Pfau

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In the last decade, soft-X-ray holography particularly found application in nanoscale imaging of magnetic samples [1]. As image contrast, the magnetic dichroism in the absorption of circularly polarized X-rays (XMCD effect) is exploited that allows to element-selectively probe different constituents, e.g., different layers, of the sample. Due to the integration of the X-ray holography optics and the sample into a single compact unit, the imaging process is inherently drift-free and the samples can be readily investigated under different environmental conditions such as magnetic field or temperature. Here, I will present a study on samples where a magnetic multilayer is patterned into islands of 80nm width—a prototype for bit-patterned media [2]. Taking advantage of the possibility of X-ray holography to simultaneously image different samples, we systematically studied the magnetic switching of individual islands within patterns supporting different bit densities in order to identify origins for the island-to-island variation of the switching field.

References:

[1] B. Pfau, S. Eisebitt, X-Ray Holography. In: Synchrotron Light Sources and Free-Electron lasers, 1st edn. (Springer, Berlin, 2015), doi: 10.1007/ 978-3-319-14394-1_18-1 (in print)
[2] B. Pfau et al., APL **99**, 062502 (2011), B. Pfau et al., APL **105**, 132407 (2014)

Abstracts of the PEEM Session

Monday, 5th of October

Chair: Ernst Bauer

Location: BESSY II Lecture hall

Cathode Lens Spectromicroscopy With Polarized X-Rays

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Cathode lens electron microscopy has taken a breathtaking evolution over the last decades – thanks to the increased availability of synchrotron radiation. The combination of wide spectral tunability, variable light polarization and well-defined time-structure has led to a broad range of applications in solid state physics and materials science. Recent years have witnessed new instrumental developments, particularly with respect to spectromicroscopy applications using hard x-rays [1], electron momentum mapping [2] and spin resolution [3].

In this contribution we will discuss some results obtained with these new spectromicroscopy approaches. The excitation with hard x-rays offers a higher information depth of photoelectrons, which is employed for the study of the chemical changes occurring in memristive oxide systems during electrical switching. The electron momentum mapping or “k-space microscopy” is used to study the absorption behavior and geometry of molecules on surfaces. The power of spin-resolved k-space microscopy is demonstrated for ferromagnetic Cobalt films, yielding a very efficient band mapping. We will also consider the future developments at BESSY, which may open up new opportunities for cathode lens spectromicroscopy.

References:

[1] C. Wiemann, et al., Appl. Phys. Lett. **100**, 223106 (2012).
[2] A. Winkelmann, et al., New J. Phys. **14**, 18 (2012).
[3] C. Tusche, et al., Ultramicroscopy **130**, 70 (2013).

Studying magnetism at the nanoscale using X-PEEM at the SLS

Armin Kleibert

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Magnetism at the nanoscale is a vibrant research field in condensed matter physics, which is not only driven by the continuing demand for miniaturization of electronic and magnetic devices, but also by the unique properties and phenomena that emerge in nanomagnets due to spatial confinement and the high surface-to-volume ratio. Over the last two decades X-ray photo-emission electron microscopy (X-PEEM) has proven to be a powerful platform to study magnetic phenomena at the nanoscale and has contributed significantly to our current understanding of nanomagnetism. This success is due to its unique capabilities to provide chemical, electronic and magnetic information in a spatially and time-resolved manner with element-specific sensitivity to the smallest amounts of matter [1].

At the surface/interface: microscopy (SIM) beamline at the Swiss Light Source (SLS) a broad research program that includes in-house and user projects benefits from the unique features of X-PEEM [2]. The investigations span from molecular magnetism at interfaces to artificial spin ice and laser-induced magnetization dynamics. In this contribution, we will highlight some recent studies performed at the SIM beamline with emphasis on magnetic nanoparticle systems. In particular, we will show that X-PEEM can reveal unexpected magnetic metastability in 3d transition metal nanoparticles with sizes down to 10 nm and less [3]. These findings not only help to clarify large discrepancies found in the literature, but provide also important insights to our understanding of the size-dependent evolution of magnetic properties in condensed matter. Moreover, our results may open new ways to tuning the properties of nanomagnets for applications.

References:

[1] A. Locatelli and E. Bauer, J. Phys. Condens. Matter, 20, 82202 (2008)
[2] L. Le Guyader *et al.*, J. Elec. Spectr. Rel. Phen., 185, 371 (2012)
[3] A. Balan *et al.*, PRL, 112, 107201, (2014)

Recent Developments in Cathode Lens Microscopy

Rudolf M Tromp

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2 Leiden Institute of Physics, Kamerlingh Onnes Laboratory, Leiden University, The Netherlands

Cathode lens microscopy, comprising both Photo Electron Emission Microscopy (PEEM) and Low Energy Electron Microscopy (LEEM), has undergone major developments over the last decade. This includes correction of spherical and chromatic aberration, the increasing use of PEEM for spatially resolved k-space imaging of occupied electron bands, and –most recently- the use of LEEM for spatially resolved k-space imaging of un-occupied electron bands. Correction of spherical and chromatic aberration is now becoming more broadly available, and had distinct advantages for use in synchrotron-based PEEM. However, setup of optimum imaging conditions for a given experiment is still open to discussion. I will show how the electron mirror optics, in combination with the objective lens, can be configured as an *adjustable* achromat. For example, an achromat centered around a start energy of 2.5 eV, with a bandwidth of 5 eV, yields a spatial resolution of 4-5 nm, while an achromat centered at 30 eV, with a passband from 9 to 62 eV has a resolution of 15 nm. Such a very wide passband (resulting in high transmission) may be extremely useful for imaging samples with weak signals, as often encountered in practice. I will also discuss the prospects for developing an apochromatic system, which would further improve resolution by another factor 2x, and transmission by a factor 10x. Finally, I will show recent results on measuring un-occupied bandstructures by LEEM, yielding information complementary to traditional ARUPS experiments.

Nanopaleomagnetism of iron and stony iron meteorites: new methods, new insights

Richard Harrison[1]

1 Department of Earth Sciences, University of Cambridge, U.K.

Microstructural and geochemical studies of meteoritic metal have been instrumental in shaping our current views of differentiated planetesimals, providing constraints on their cooling rate, their size, the timing of their differentiation and their fractional crystallization and impact histories. The characteristic Widmanstätten microstructure, familiar to anyone who has looked at a polished and etched section of an iron meteorite with the naked eye, hides a nanoscale complexity that is revealed only with high-resolution electron microscopy – a legacy of stranded diffusion profiles, metastability, martensitic transformations, chemical segregation and ordering during slow cooling over millios of years on the parent body. The presence of soft bcc iron has traditionally lead to the meteoritic metal being dismissed as a reliable carrier of paleomagnetic information. However, we have shown that, under favorable circumstances, paleomagnetic information can be recorded and retained on a local scale within a unique nanoscale intergrowth called the cloudy zone (CZ).[1] High-resolution XPEEM enables the magnetic state of the CZ to be imaged and analyzed quantitatively, opening up new avenues of research into the nanopaleomagnetism of a range of meteorites. Such studies are not only revealing new insight into the thermochemical properties of planetesimals in the early solar system, but provide us with unique opportunities to learn about how magnetic fields are generated on planetary bodies in general, and the underlying physics of the dynamo generation process itself.

References:

1. Nature 517: 472-475

Abstracts of the TXM Session

Tuesday, 6th of October

Chair: Eva Pereiro

Location: BESSY II Lecture hall

Nanoscale NEXAFS analysis of individual N-doped TiO_2 nanoribbons

C. Bittencourt[1], M. Rutar[2,3], P. Umek[2], A. Mrzel[2], K. Vozel[2], D. Arčon[2,4], P. Guttmann[5]

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2 Jožef Stefan Institute, Ljubljana, Slovenia
3 Jožef Stefan International Postgraduate School, Ljubljana, Slovenia
4 Faculty of Mathematics and Physics, University of Ljubljana, Slovenia
5 HZB, Institute for Soft Matter and Functional Materials, Berlin, Germany

Nitrogen doping titanium dioxide (TiO_2), with N at substitutional sites has been reported to be indispensable for enhancing the use of TiO2 as a visible-light photocatalytic material. The main effect of the N-doping is the narrowing of the energy band gap of TiO_2 due to the mixing of N 2p and O 2p states. Additionally, an isolated narrow band responsible for the visible light photoactivity is formed above the valence band. Nitrogen doping into the TiO_2 lattice is rather challenging as N atoms must be accommodated geometrically and electronically. To address the fundamental issues we explored a chemical route and report here a comprehensive investigation of N-doped TiO_2 nanoribbons. As a precursor material for N-doped TiO_2 nanoribbons we used hydrogen titanate nanoribbons (HTiNRs) because upon heating HTiNRs easily transform first to the monoclinic TiO_2 β-phase (TiO_2-B) and then to anatase (tetragonal phase) while retaining the morphology of the parent nanostructure intact.

The nitrogen doping of TiO_2 nanoribbons during the thermal transformation of hydrogen titanate nanoribbons (HTiNRs) between 400 and 800 °C in a dynamic ammonia atmosphere was investigated using X-ray photoelectron spectroscopy (XPS) and transmission X-ray microscopy combined with near-edge X-ray absorption fine structure spectroscopy (NEXAFS-TXM). XPS results clearly reveal the nitrogen doping of TiO_2 nanoribbons and that, depending on the calcination temperature, nitrogen atoms occupy interstitial and substitutional sites. Moreover, in samples calcined at 580 and 650 °C the presence of N_2-like species in the HTiNRs was detected by NEXAFS-TXM. These species are trapped in the HTiNRs structure.

References:

Beilstein J. Nanotech. 6,831,(2015)
Beilstein J. Nanotech. 3,789,(2012)
RSC Adv. 5, 23350, (2015)
Nature Photonics 6,25,(2012)

Correlative cryo-fluorescence and cryo-soft X-ray tomography of autophagy in whole, unstained mammalian cells

MC Domart[1], R Carzaniga[1], M Razi[2], SA Tooze[2], E Duke[3], LM Collinson[1]

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3. Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxon, UK.

Cryo-soft X-ray tomography (cryo-SXT) is a synchrotron-hosted imaging technique used to analyse the ultrastructure of intact, cryo-prepared cells. Correlation of cryo-fluorescence microscopy and cryo-SXT can be used to localise fluorescent proteins to organelles preserved close to native-state. Cryo-correlative light and X-ray microscopy (cryo-CLXM) is particularly useful for the study of organelles that are susceptible to chemical fixation artefacts during sample preparation for electron microscopy. In our recent work, we used cryo-CLXM to characterise GFP-LC3-positive early autophagosomes in nutrient-starved HEK-293A cells (Duke et al., 2013). Cup-shaped omegasomes were found to form at ‘hot-spots’ on the endoplasmic reticulum. Furthermore, cryo-SXT image stacks revealed the presence of large complex networks of tubulated mitochondria in the starved cells, which would be challenging to model at this scale and resolution using light or electron microscopy.

Other biological application examples will be discussed with intent to give a flavour of the kind of study that would benefit from this novel imaging platform, though applications will undoubtedly expand as more cell biologists adopt the technique.

References:

Ultramicroscopy, 143:77-87 (2014)
Protoplasma, 251(2):449-58 (2014)
J. Microscopy, 255 (2):65-70 (2014)
Methods Cell Biol, 124:151-78 (2014)

X-ray microscopy and optics development at HZB

S. Werner[1], P. Guttmann[1], S. Rehbein[1], C. Pratsch[1], J. G. McNally[1], G. Schneider[1]

1 Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Institute for Soft Matter and Functional Materials, Albert-Einstein-Str. 15, 12489 Berlin, Germany

During the last three decades X-ray microscopy (XM) has been established as a powerful imaging technique for investigations on the nanoscale. A wide range of applications from material, energy, environmental and life science benefits from the unique capabilities given by XM. This method combines high resolving power in the nanometer range with the feasibility to image thick samples due to the high penetration depth of X-ray photons.

At the Helmholtz-Zentrum Berlin (HZB) we operate a full-field transmission X-ray microscope (TXM) in the soft X-ray photon energy range at an undulator beamline [1]. The specialized optical setup of the current HZB-TXM permits spectromicroscopic applications combining high spectral resolution and structural information as well as X-ray nano-tomography of cryogenic samples [2,3]. Thereby, the achievable spatial resolution of the TXM is limited by the numerical aperture of the zone plate objective and the wavelength used for imaging. Understanding of the image formation is crucial to visualize nanoscale sample details. We will discuss the X-ray imaging performance of the HZB-TXM in terms of resolution and efficiency and present recent nanotechnological results regarding the in-house zone plate fabrication [4,5].

Our future work focuses on the development of new techniques and advanced optical setups to overcome current limitations in lens-based X-ray imaging [6]. We will report on new approaches to increase the 2D and 3D resolution for soft X-ray microscopy and discuss a novel nanofabrication technique which has the potential to manufacture high efficient, high resolution soft and hard X-ray optics.

References:

[1] S. Heim, et al., Journal of Physics: Conference Series 186, 012041 (2009)
[2] P. Guttmann, et al., Nature Photonics 6, 25-29 (2012)
[3] G. Schneider, et al., Nature Methods 7, 985-987 (2010)
[4] S. Rehbein, et al., Optics Express 20, 5830-5839 (2012)
[5] S. Werner, et al., Nano Research 7, 528–535 (2014)
[6] G. Schneider, et al., J. Struct. Biol. 177, 212-223 (2012)

Abstracts of the Ptychography / STXM Session

Tuesday, 6th of October

Chair: Chris Jacobson

Location: BESSY II Lecture hall

Scanning imaging employing phase and scattering-based contrast

Oliver Bunk

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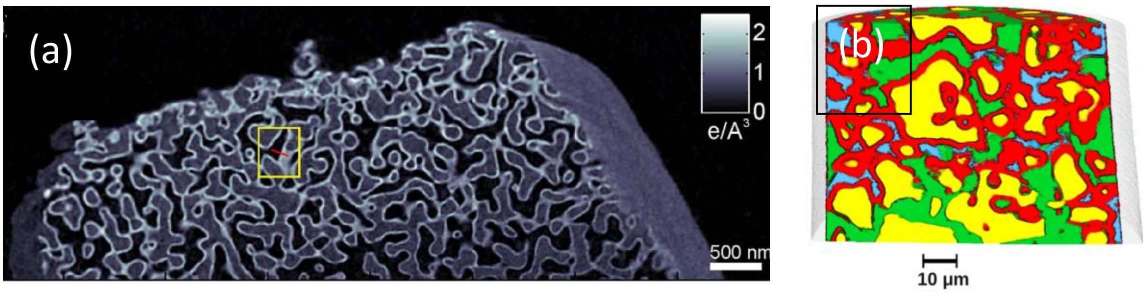
The field of X-ray imaging has seen tremendous developments in the past decade. Phase contrast is regularly utilized at storage ring and lab-based sources and a number of methods link spectroscopy, scattering, and diffraction with imaging. Techniques like Talbot interferometry, scanning transmission X-ray microscopy (STXM), ptychographic coherent diffraction imaging (PCDI) and scanning small-angle X-ray scattering (scanning SAXS) are both closely related and complementing each other. In this presentation the status at the Swiss Light Source (SLS) with the focus on PCDI and scanning SAXS will be presented and hints will be given on the profound impact future diffraction-limited light sources might have for these techniques and their applications.



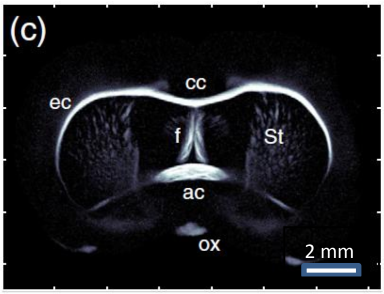


Figure: (a) With ptychographic coherent diffraction imaging 16 nm isotropic resolution in 3D have been demonstrated on a test sample [1]. (b) This method is for example utilized for the study of catalysts [2]. (c) With scanning small-angle X-ray scattering nanoscale properties like the diffraction from myelin sheaths can be imaged over macroscopic length scale.

References:

[1] M. Holler, A. Diaz, M. Guizar-Sicairos, P. Karvinen, E. Färm, M. Härkönen, M. Ritala, A. Menzel, J. Raabe, and O. Bunk, Scientific Reports **4**, 3857 (2014).
[2] J.C. da Silva, P. Trtik, A. Diaz, M. Holler, M. Guizar-Sicairos, J. Raabe, O. Bunk, and A. Menzel, Langmuir **31**, 3779-3783 (2015).
[3] T.H. Jensen, M. Bech, O. Bunk, A. Menzel, A. Bouchet, G. Le Duc, R. Feidenhans'l, and F. Pfeiffer, NeuroImage 57, 124-129 (2011).

Cryo X-ray Tomography: 3D cellular ultrastructure without chemical fixation or staining

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A particular advantage of soft X-ray microscopy is that it enables 3D nanoscale imaging of intact biological specimens with thicknesses up to 10 um. Cryo-preservation is performed to prevent damage by the X-ray beam, but otherwise specimens can be examined in their native state, that is without any form of chemical fixation or chemical staining. Contrast within specimens is obtained by using an X-ray wavelength of 2.4 nm in which organic material absorbs much more strongly than water. 3D images of intact cells are generated by using tomography, namely by acquiring a series of images from the specimen at different tilt angles from -60° to +60°, and then applying standard tomographic algorithms to generate a 3D reconstruction. The resultant 3D images of intact eukaryotic cells have achieved resolutions in the range from 30-70 nm. This enables clear-cut 3D visualization of all membrane-bound ultrastructures, including the plasma membrane, mitochondria and their cristae, endoplasmic reticulum, Golgi, intracellular vesicles, double nuclear membrane with nuclear pores and nuclear membrane channels. Other sub-cellular structures such as microtubules and nucleoli can also be discerned. These capabilities fill an important niche in biology, as conventional electron microscopy can only obtain thin slices (~50 nm) of chemically preserved, dehydrated specimens. Cryo-electron microscopy like X-ray microscopy, can also examine unfixed, near-native specimens, but it is limited to thicknesses of only 1 um, far less than the typical size of a eukaryotic cell. The current X-ray microscope at the Helmholtz Zentrum also enables correlative fluorescence and X-ray imaging, providing a valuable tool for 3D ultrastructural localization of either antibody or fluorescent-protein marked sub-cellular structures. Note that the current resolution limit of 30 nm in biological structures is not set by the X-ray wavelength of 2.4 nm, but rather by the optics required to focus the X-rays and also by approximations made to simplify the reconstruction process of a thick specimen. Thus higher resolution X-ray images are possible with a realistic goal of 10 nm in x,y and z. Some of these future possibilities will be discussed.

Advanced Scanning X-ray Microscopy at the MAXYMUS Beamline

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MAXYMUS is a unique Scanning Transmission X-ray Microscope (STXM) operated by the Max Planck Institute for Intelligent Systems at the Bessy II synchrotron in Berlin, Germany, and a world leading instrument in particular for magnetization dynamics imaging. It combines established STXM properties like high spatial and energy resolution [1] with unique features like a highly optimized time resolved measurements system and UHV capability, utilizing combined surface sensitive (total electron yield) and bulk sensitive (transmission mode) detection methods [2]. In addition zone plate scanning is available for extremely fast scanning speeds and the use of complex or bulky sample assemblies.

We will present examples of recent research at MAXYMUS by both MPI [3] and external users [4] and demonstrate the potential of the system for high level research on magnetic devices. We also give an outlook on future developments, which are in the status of installation and testing, which will dramatically increase our possibilities. Special highlights are the development of a helium sample cryostat, a newly acquired fast X-ray CCD for ptychographic imaging, and the possibilities for sub 10ps time resolved imaging made possible by the future BESSY VSR upgrade.

References:

[1] C. Pölker et al., Science 337, 1075-1078 (2012)
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Abstracts of the µm Tomography / Multiprobe Scanning Imaging Session

Tuesday, 6th of October

Chair: Peter Fratzl

Location: BESSY II Lecture hall

Game-changing technologies: the impact of list-mode data acquisition on x-ray fluorescence microscopy at the Australian Synchrotron

Martin de Jonge[1], David Paterson[1], Daryl Howard[1], Nader Afshar[1], Robin Kirkham[2], Chris Ryan[2]

1 Australian Synchrotron, Australia
2 CSIRO, Australia

The Australian Synchrotron's X-Ray Fluorescence Microprobe operates at a length / intensity scale 1-3 orders of magnitude poorer than current leading microprobes, and ~4-6 orders of magnitude poorer than anticipated following DLSR upgrades [1]. Despite this, our adoption and optimisation of the Maia detector system has resulted in a number of novel developments, including XRF tomography, XANES imaging, and large-area mapping on a routine basis, and XANES tomography as a developmental technology.

How? The Maia detector system comprehensively addresses three limitations of present-generation x-ray fluorescence detection: (1) improved experimental efficiency through increased detector solid angle; (2) high overall count-rate through the use of 384 parallel detectors, and; (3) the use of list-mode data acquisition. While (1) and (2) address detector and experimental efficiency (and might be met using other approaches), it is the transition to list-mode data acquisition, including hardware integration of sample motion events with detector events, that enables us to achieve these exotic measurement modalities.

We discuss the impact of the Maia detector system's list mode operation on the beamline measurement system, and reflect on a fundamental shift in our conception of the measurement limitations. We show evolution of both beamline and detector integration toward a purpose-engineered system has delivered increasing performance. We describe some future developments designed to further optimise the power of this measurement system, to address a range of user science applications. These concepts are translated to next-generation storage rings, with a view to understanding possibilities for future measurement modalities.

References:

[1] de Jonge, Ryan & Jacobsen, J. Synchrot. Rad. **21** (2014).

Synchrotron X-ray Imaging in Energy Research

I. Manke[1], A. Hilger[1], T. Arlt[1], H. Markötter[1], N. Kardjilov[1], F. Sun[1], S. Risse[1], J. Scholta[2], W. Lehnert[3], H. Riesemeier[4], V. Schmidt[5], J. Banhart[1]

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3 Forschungszentrum Jülich (FZJ), Germany
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5 University of Ulm, Germany

In recent years research on energy-related materials became one of the most important application fields of synchrotron X-ray imaging. Most materials used for energy storage and conversion have a complex three-dimensional structure and morphology on the nano- and micrometer scale, knowledge of which is important for both understanding the function of materials and energy conversion systems and for their further development. Radiographic and tomographic measurement techniques based on synchrotron X-rays are ideally suited to obtain detailed information not only on material structures. These techniques are (if used carefully) non-destructive and allow for in-operando investigations of structural changes and media distributions. This talk provides an overview to recent developments on the field of in-operando and ex-situ radiography and tomography on batteries [1-3], electrolyzers [4] and fuel cells [5] at Helmholtz Centre Berlin for Materials and Energy (HZB). It will be demonstrated how information contained in X-ray absorption edges, like XANES/EXAFS features, can be extracted for analysis of electrode materials [6]. Furthermore an example will be given, that demonstrates how three-dimensional imaging can significantly contribute to more realistic modelling and simulations [7].

References:

[1] Appl. Phys. Lett., 90, 214102 (2007),
[2] Adv. Energy Mat., 5, 5, 1401612 (2015)
[3] Scientific Reports, 8, 11 (2015)
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[5] Electrochem. Comm. 51, p. 133 (2015)
[6] J. Power S. 221, p. 210 (2013)
[7] J. Power S. 257, p. 52 (2014)

Full-field X-ray orientation microscopy

Wolfgang Ludwig[1,2], Nicola Vigano[1,2], J. Batenburg[3]

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X-ray diffraction contrast tomography [1,2] is a near-field X-ray diffraction imaging technique enabling simultaneous characterization of the 3D grain microstructure in polycrystalline materials fulfilling conditions in terms of grain size and intra-granular orientation spread. With the recent upgrade to a six-dimensional reconstruction framework [3,4] the method now provides access to spatially resolved crystal orientation maps with an orientation resolution comparable to EBSD and a spatial resolution consistent with the voxel size of the high resolution X-ray imaging detectors.

Moreover, like modern electron microscopes, state of the art X-ray diffraction imaging instruments can nowadays offer complementary imaging modalities like phase contrast tomography, X-ray topography and dark-field microscopy. This in turn provides unique possibilities for non-destructive characterization of 3D microstructures and their evolution as a function of applied strain or temperature. We will illustrate the current possibilities and limitations of combined X-ray diffraction imaging experiments with observations acquired during the onset of plastic deformation in miniature tensile and compression specimen made from Al and Ti alloy.

References:

[1] Rev. Sci. Instr. **80**, (2009)
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[3] J.Appl.Cryst. **47**, 1826 (2014)
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Poster Abstracts

Monday, 5th of October, BESSY II Foyer

IN-OPERANDO SYNCHROTRON X-RAY RADIOGRAPHY FOR HT-PEFC RESEARCH

T. Arlt, W. Lüke, C. Wannek, H. Markötter, J. Banhart, W. Lehnert, D. Stolten, I. Manke
Helmholtz-Zentrum Berlin für Materialien und Energie

In high temperature polymer electrolyte fuel cells (HT-PEFCs) the proton conductivity relies on phosphoric acid doped polybenzimidazole as electrolyte. Up to now research activities on HT-PEFCs to analyze the distribution of phosphoric acid within the MEA were limited to averaging and ex-situ methods. For the first time we were able to combine synchrotron X-ray radiography with electrochemical impedance spectroscopy to investigate different fuel cells at different operating conditions.

2 In situ µ-Tomography investigations of creep damage of Lightmetal based MMCs

B. Camin, A. Kilian, D. Souza, W. Reimers
Technische Universität Berlin

The creep damage mechanisms in Lightmetal based Metal Matrix Composites (MMCs) reinforced with particles as well as hybrid with particles and fibers are analysed. In situ µ-tomography during creep enables time-resolved and space-resolved detection of the creep damage. The microstructural damage mechanisms such as void formation in the matrix, reinforcement fracture and delamination of the matrix from the reinforcements can be detected and correlated to the macroscopic creep curve.

3 Visualizing Reaction and Transport Processes in Metal-Air-Batteries via X-ray Tomography

Daniel Schröder, Conrad L. Bender, Andre Hilger, Tobias Arlt, Markus Osenberg, Ingo Manke, Jürgen Janek
Physikalisch-Chemisches Institut, AG Janek, Justus-Liebig-Universität Giessen

Currently, a wider understanding of fundamental processes in metal-air-batteries is needed to promote them as next generation batteries. In this work we use in-situ and ex-situ X-ray tomography to show the local distribution of reaction products in various metal-air-batteries (zinc-air- and sodium-oxygen-battery). The analysis yields crucial information for their operation strategy and can potentially be applied to understand processes in other next generation batteries.

4 Characterization of hydrogen assisted cracking using synchrotron refraction computed tomography

Rene Laquai, Thomas Schaupp, Bernd R. Mueller, Axel Griesche, Giovanni Bruno, Thomas Kannengiesser
BAM Federal Institute for Materials Research and Testing

We investigated the hydrogen-induced crack formation in metallic alloys under uniaxial tensile loading by synchrotron refraction computed tomography (SRCT) at the BAMline at BESSY. This allows to gain information about the size and distribution of cracks with a better resolution than the X-ray computed tomography (CT). Preliminary results on aluminum alloys show that this technique allows to resolve the full 3D structure of cracks, which is undetected by absorption based CT.

5 Understanding the composition of marine adhesives by X-Ray microanalysis

S. Stuhr, T. Senkbeil, A. Rosenhahn, Ruhr-Universität Bochum Y. Yang, P. Cloetens, ESRF, Grenoble D. Batchelor, R. Simon, ANKA, KIT G. Falkenberg, PETRA III, DESY
Ruhr-Universität Bochum

For an efficient prevention of biofouling it is important to understand the process of adhesion and thus the composition of the adhesives. Important fouling organisms are barnacles and the diatom Navicula perminuta. X-ray microanalysis reveals elemental distributions in the underwater adhesives and the data obtained at different synchrotron sources is discussed on the background of the organic compounds involved.

6 Polymer Electrolyte Membrane Fuel Cells visualization using Synchrotron x-ray radiography

Saad S. Alrwashdeh , H. Markötter,T. Arlt , J. Haussmann, M. Klages, J. Scholta, I. Manke, J. Banhart
Helmholtz-Zentrum Berlin für Materialien und Energie

Water transport in PEMFCs is investigated by synchrotron radiography as a non-destructive testing method. Two perspectives are described on this poster, namely in-plane and through-plane, which yield information on the water distribution in the different cell materials. It is shown how the water distribution and transport is quantified and visualized. With this method investigations of different cell materials at different operating parameters are conducted.

7 Local electrical control of the Ferromagnetic/Antiferromagnetic transition in FeRh just above room temperature

S. Valencia, L. C. Phillips, A. A. Ünal, F. Kronast, R. O. Cherifi, V. Ivanovskaya, A. Zobelli, I. C. Infante, E. Jacquet, N. Guiblin, B. Dkhil, A. Barthelemy, M. Bibes.
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By means of X-Ray PEEM we show the possibility to locally switch by means of a voltage between the FM and AFM phase of epitaxial FeRh thin films grown on top of ferroelastic BaTiO3. The resultant magnetoelectric coupling is larger and more reversible than previously reported from macroscopic measurements. Our results emphasize the importance of nanoscale ferroic domain structure and the promise of first-order transition materials to achieve enhanced coupling in artificial multiferroics.

8 Energy selective quasi-simultaneous imaging and “energy loss” electron momentum mapping in DEEM

Krzysztof P. Grzelakowski
OPTICON Nanotechnology

We report the first test measurements carried out with the newly developed technique Dual EEM. It utilizes a novel concept of the electron sample illumination with in-lens electron gun geometry that enables alongside the energy selective secondary electron imaging and secondary/inelastic (energy loss) electron momentum mapping.As a test object a Cs grown on the Mo(110) surface has been chosen for the energy selective quasi-simultaneous observations in the real as well as in the reciprocal plane.

9 X-PEEM investigation of chemical and electronic surface properties of solution processed perovskite-based thin-film solar cell structures

C. Hartmann, G. Sadoughi, R.G. Wilks, H. Klemm, G. Peschel, E. Madej, A. Fuhrich, E. Handick, S. Raoux, Th. Schmidt, H. Snaith, and M. Bär
Helmholtz-Zentrum Berlin für Materialien und Energie

X-PEEM investigation provides insight into coverage, morphology, and local elemental composition of the interface of 300 nm CH3NH3PbI(3-x)Clx perovskite thin films on planar compact TiO2 on FTO/glass substrates. We find incomplete coverage, with holes reaching to the compact TiO2 substrate. In both covered areas and holes we find only a single Pb species; some areas of more complex Pb chemistry have an additional lower binding energy species.

10 Low Temperature Scanning X-Ray Microscopy of Supercurrents in YBCO

Claudia Stahl, Stephen Ruoss, Markus Weigand, Michael Bechtel, Gisela Schütz, Joachim Albrecht
Max Planck Institute for Intelligent Systems, Stuttgart

Based on the magnetic interaction between the stray fields of a current carrying superconductor and the magnetization of an adjacent ferromagnet, scanning X-ray microscopy based on the XMCD effect can reveal the distribution of the flux line density within the superconductor with high spatial resolution. We present XMCD microscopy images of the magnetic flux density and current distribution in YBCO obtained with the TEY mode in the low temperature setup in the SXM MAXYMUS at BESSY II in Berlin.

11 Resonant soft X-ray ptychography with the HORST chamber

C. Rumancev, S. Stuhr, A. von Gundlach, T. Gorniak, T. Senkbeil, A. Rosenhahn
Analytical Chemistry - Biointerfaces, Ruhr-University Bochum

The holographic X-Ray scattering apparatus HORST is a vacuum chamber developed for scattering experiments at synchrotron and free-electron laser sources. Recent upgrades enable soft X-ray ptychography to image nonperiodic objects at high spatial resolution. Resonant experiments provide chemical information which can be exploited for frozen-hydrated biological specimen using a cryogenic sample environment.

12 The nucleus at a glance – what soft X-ray cryo-microscopy/tomography can do for structural cell biology

Christoph Hagen, Kay Grünewald
University of Oxford, Wellcome Trust Centre for Human Genetics, STRUBI/OPIC

How about an imaging technique providing you with absorption-contrast based three-dimensional structural information at a resolution of ~30 nm, from best-possible preserved biological objects up to 12 µm thick, which can be spatially correlated with specific information from fluorescence cryo-microscopy? That this is not only a dream anymore will be shown along our results on herpesvirus-induced changes of the nuclear ultrastructure.

13 Imaging intracellular calcium accumulation in coccolithophores using cryo-X-ray tomography

Assaf Gal, Andrea Sorrentino, Eva Pereiro, Damien Faivre, Andre Scheffel
Max-Planck Instittue of Colloids and Interfaces

Coccolithophore algae produce minute calcitic scales inside intracellular compartments. We used soft-X-ray tomography of cryo-preserved cells in order to study the calcium pathway in the cell. The reconstructed data revealed intracellular compartments packed with absorbing material. Spectro-microscopy of the cells at the Ca L-edge showed that this is a disordered Ca-rich phase. These findings suggest that intermediate stages are part of the calcium accumulation process during calcification.

14 BESSY VSR – The Variable pulse length Storage Ring - From BESSY II to BESSY VSR

Paul Goslawski, Andreas Jankowiak, Jens Knobloch, Axel Neumann, Markus Ries, Martin Ruprecht, Adolfo Velez, Godehard Wüstefeld
Helmholtz-Zentrum Berlin für Materialien und Energie

BESSY VSR, a scheme where 1.7 ps and 17 ps long electron bunches (rms) can be stored simultaneously in the storage ring, is the major upgrade programme for BESSY II. This poster explains the basic principles and discusses the improvements for user operation.

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Oliver Bunk (PSI)
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Peter Fratzl (MPI Golm)
Richard Harrison (University of Cambridge)
Chris Jacobson (Argonne Lab/Northwestern University)
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Martin de Jonge (Australian Synchrotron)
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Meeting Venue

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