KFM 10: Spectroscopy and Microscopy I with X-rays and Ions

Chair: Burkhard Beckhoff - Physikalisch-Technische Bundesanstalt, Berlin, Germany

Time: Tuesday 9:30–13:40 Location: EMH 225

Invited Talk KFM 10.1 Tue 9:30 EMH 225

Insights into the Inside provided by Coherent X-ray Imaging — •Tim Salditt — Georg-August-Universität Göttingen, Institut für Röntgenphysik, Friedrich-Hund-Platz 1, 37077 Göttingen

X-rays can provide information about the functional architecture of materials, including soft and biological matter [1 ,2], also in a heterogeneous state and in operando [3]. However, the specific advantages of hard x-rays in view of penetration power, high spatial resolution, quantitative contrast, and compatibility with environmental conditions is to date not fully exploited, mainly due to shortcomings in x-ray optics. With the advent of highly brilliant radiation, coherent focusing, and lens-less diffractive imaging this situation has begun to change. Nanofocused coherent x-ray synchrotron beams are now routinely used for scanning as well as for full field holographic x-ray imaging, in 2d and 3d. Even 4d movies of materials [4] are enabled by time-resolved tomography, providing new insights into the inside of materials as well as functional processes. In this talk I present the optical concepts and challenges, including phase retrieval and image reconstruction, and explain all the buss about holography, ptychography and tomography.

[1] M. Bartels, M. Krenkel, J. Haber, R. N. Wilke, and T. Salditt, Phys. Rev. Lett. (2015), 114, 048103. [2] M. Töpperwien, M. Krenkel, D. Vincenz , F. Stöber, A. M. Oelschlegel, J. Goldschmidt and T. Salditt, Scientific Reports (2017), 7, 42847. [3] J. Wallentin, M. Osterhoff and T. Salditt, Adv. Mater. (2016), 28, 1788. [4] A. Ruhlandt, M. Töpperwien, M. Krenkel, R. Mokso, and T. Salditt, Scientific Reports (2017), 7, p. 6487.

KFM 10.2 Tue 10:00 EMH 225

Laboratory soft X-ray Tomography — •Aurélie Dehlinger[1,2], Julia Bränzel[1,3], Daniel Grötzsch[1,2], Robert Jung[3], Birgit Kanngiesser[1,2], Stefan Rehbein[4], Christian Seim[5], and Holger Stiel[1,3] — [1]Berlin Laboratory for innovative X-ray technologies (BLiX) — [2]Technische Universität Berlin, Institut für Optik und Atomare Physik, Hardenbergstr. 36, 10623 Berlin, Germany — [3]Max-Born-Institut, Max-Born-Str. 2A, 12489 Berlin, Germany — [4]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Albert-Einstein-Straße 15, 12489 Berlin, Germany — [5]Physikalisch-Technische Bundesanstalt, Abbestr 2-12, 10587 Berlin, Germany

In microscopy, where the resolution depends on the wavelength of the probing light, soft X-rays can be used to analyze samples that cannot be resolved with visible light microscopes. X-ray microscopy in the water window allows imaging with resolutions in the nanometer regime as well as a high contrast between carbon and oxygen, which is an ideal condition for the tomographic investigation of biological samples in their natural state. We present a Full-Field Laboratory Transmission X-ray Microscope (LTXM) at the Berlin Laboratory for innovative X-ray technologies (BLiX) with a probing radiation energy of 500 eV, provided by a laser-based nitrogen plasma source. The development of laboratory X-ray sources aims to increase the availability of X-ray microscopy to a broader scientific community. We will present the latest measurements carried out on biological samples and the corresponding reconstructed tomograms, which are the key to a more precise and global analysis in various fields of life science.

KFM 10.3 Tue 10:20 EMH 225

Tomography with extended sources — •Leon Merten Lohse, Malte Vassholz, and Tim Salditt — Institut für Röntgenphysik, Friedrich-Hund-Platz 1, 37077 Göttingen, Deutschland

X-ray computed tomography (CT) is widely used today for nondestructive 3D imaging. In practice, the achievable resolution for analytical CT in the laboratory is still limited by the low brilliance of table top sources. One way to overcome this limitation is the use of a line source, as has been demonstrated recently [2]. The larger x-ray source spot allows more photons to contribute to image formation. It has been shown that a reconstruction scheme based on the 3D Radon transform is in fact compatible with a line source and the impact on the resolution can be controlled to be minor [1]. As the method allows to increase the flux at constant resolution, it promises to overcome the trade-off between acquisition time and resolution, and ultimately to allow higher resolutions in laboratory-based analytical CT.

[1] L. M. Lohse et al. ”Tomography with extended sources: ...”, Phys. Rev. A (accepted 11/2017) [2] M. Vassholz et al. ”New X-Ray Tomography Method ... ”, Phys. Rev. Lett. (2016)

KFM 10.4 Tue 10:40 EMH 225

Imaging with Nanometer Resolution from 8 to 100 keV using Multilayer Zone Plates (MZP) — •Jakob Soltau, Tim Salditt, and Markus Osterhoff — Institut für Röntgenphysik, Georg-August-Universität Göttingen, Göttingen, Germany

The brilliance of modern synchrotrons cleared the path towards generating highly focussed x-ray beams. Latest developments in the fabrication of multilayer zone plates (MZP) using the pulsed laser deposition process have enabled to decrease the resolution defining smallest zone widths down to 5 nm [1]. At the same time the optical thickness, important for efficiency, was enlarged up to 30 μm. In recent experiments at Petra III endstation P10, the MZPs were used in a scanning microscopy setup at 13.8 keV. It was possible to resolve a Siemens star test pattern with smallest feature sizes of 50 nm. A first proof-of-concept experiment at the ESRF endstation ID31, extended the limit of imaging with MZPs to photon energies of 100 keV [2].

[1] C. Eberl et al. Fabrication of laser deposited high... In: Applied surface science 307 (2014)

[2] M. Osterhoff, et al. , Ultra-high-aspect multilayer zone plates for even higher x-ray energies, Proc. SPIE (2017)

KFM 10.5 Tue 11:00 EMH 225

Divide and Update: Towards Single-Shot Object and Probe Retrieval for Near-Field Holography — •Johannes Hagemann[1,2] and Tim Saltditt[1] — [1]Institut für Röntgenphysik, Friedrich-Hund-Platz 1, 37077 Göttingen — [2]current address: DESY, X-Ray Nanoscience and X-Ray, Optics, Notkestraße 85, 22607 Hamburg

We present a phase reconstruction scheme for X-ray near-field holographic imaging based on a separability constraint for probe and object. In order to achieve this, we have devised an algorithm which requires only two measurements – with and without an object in the beam. This scheme is advantageous if the standard flat-field correction fails and a full ptychographic dataset can not be acquired, since either object or probe are dynamic. The scheme is validated by numerical simulations and by a proof-of-concept experiment using highly focused undulator radiation of the beamline ID16a of the European Synchrotron Radiation Facility (ESRF).

KFM 10.6 Tue 11:20 EMH 225

Multiskalige Analyse von Energiematerialien mittels Synchrotron und Focused Ion Beam — •Markus Osenberg[1], André Hilger[2], Henning Markötter[2], Tobias Arlt[1], Thomas Turek[3], Volker Schmidt[4], Joachim Binder[5], Ingo Manke[2] und John Banhart[1,2] — [1]Technische Universität Berlin — [2]Helmholtz-Zentrum Berlin — [3]Technische Universität Clausthal — [4]Universität Ulm — [5]Karlsruhe Institute of Technology

Forschung an Energiematerialien rückt immer mehr in den Fokus und gerade das Wissen über die zu Grunde liegenden, inneren Strukturen dieser Materialien, ist von entscheidender Bedeutung für die Weiterentwicklung. Energiematerialien wie zum Beispiel Batterie-, Elektrolyseelektroden oder mikroporöse Diffusionslagen aus der Brennstoffzellenindustrie wurden sowohl auf der Mikrometer als auch auf der Nanometerskala tomografiert und analysiert. Dazu wurden zum einen, hochauflösende Synchrotronmessungen am BESSY II und Focused Ion Beam (FIB) Messungen am Helmholtz-Zentrum Berlin durchgeführt. Außerdem wurden auf der Nanometerskala 2D Untersuchungen gemacht, um die Messmethoden besser miteinander verknüpfen zu können. Besonders die FIB-Messungen standen hier im Fokus bezüglich der Probenpräparation und der artefaktfreien Rekonstruktion. Auf Grundlager dieser multiskaligen Messungen konnten Zusammenhänge zwischen Herstellungsparametern und Morphologie der Materialien gezeigt werden. Darüber hinaus führten die Analysen dieser Messungen zu einem tieferen Verständnis des Rekonstruktionsprozesses von FIB-Daten und somit zu präziseren Repräsentationen der vermessenen Proben.

20 min. break

KFM 10.7 Tue 12:00 EMH 225

Simulation of Large Solid Angle Effects for XRF Quantification - First Results — •Hanna Dierks[1], Lars Lühl[1], Konstantin Andrianov[2], Thomas Wilhein[2], and Birgit Kanngiesser[1] — [1]AG Kanngießer, IOAP/TU Berlin — [2]Institute for x-optics, Hochschule Koblenz

Soft and tender X-ray microscopy (XRM) at cellular level is used worldwide to investigate biomedicine samples. Scanning transmission X-ray microscopy (STXM) in combination with fluorescence detection is able to map elements from C to Mo with a lateral resolution below 100 nm. Since biological samples generally emit rather weak fluorescence signals, a high detector efficiency (e.g large solid angle) is necessary to avoid long measurement times. The AnImaX endstation is equipped with a new annular QUAD detector which yields a very large solid angle of detection (up to 1.2 sr). Established quantification approaches normally assume a small detector area with respect to the distance sample to detector, limiting the solid angle. For large solid angles, new effects occur, since the detected radiation passes a wide angular range on its way out of the sample. This results in geometrically different exit path lengths and, for inhomogeneous samples, even different sample composition (resp. absorption coefficients). In the following, these effects are simulated based on the Sherman equation combined with an additional virtual decomposition of the sample and detector. These forward calculations aim as a first step towards the development of new quantification concepts for annular detectors with a large solid angle.

KFM 10.8 Tue 12:20 EMH 225

Scanning Transmission X-ray Microscopy with Fluorescence Detection — •Andreas Haidl[1], Konstantin Andrianov[1], Thomas Nisius[1], Lars Lühl[2], Aurelie Dehlinger[2], Hanna Dierks[2], Birgit Kanngiesser[2], and Thomas Wilhein[1] — [1]Institute for X-Optics, University of Applied Sciences Koblenz, RheinAhrCampus Remagen, Germany — [2]Institute for Optics and Atomic Physics, Technical University Berlin, Germany

X-ray microscopy can be used in many research fields for imaging applications with resolutions notable below 100nm, continuously pushing the limits to near diffraction limited resolutions.

AnImaX (Analytical Imaging with X-rays) is a flexible endstation for combining scanning and full field microscopy using synchrotron radiation in the soft X-ray spectral range. The AnImaX platform consists out of up to three separate vacuum chambers and custom designed high resolution piezo driven stages. The AnImaX endstation has been successfully put to work at the beamline P04 at PETRA III.

The use of a spatial resolved detector in scanning mode allows the simultaneous acquisition of different imaging modes by adjusting the detector response function. The CCD based multimodal detector system allows beside absorption contrast the acquisition of phase contrast and dark field images. The obtained diffraction patterns can also be used for ptychography. In the scanning mode the SDD detector with its outstanding solid angle of detection also gathers a fluorescence spectrum for each pixel. Thereby a detailed spatial elemental and chemical analysis of the specimen is performed.

KFM 10.9 Tue 12:40 EMH 225

LABORATORY(CONFOCAL) MICRO-XRF ON CRYO-FIXATED BIOLOGICAL SPECIMEN — •Frank Förste, Tobias Drechsel, Ioanna Mantouvalou, and Birgit Kanngiesser — IOAP TU Berlin, Hardenbergstraße 36, 10623 Berlin

The non-destructive imaging of elemental distributions in specimen is of high interest in many fields of research as for example in biology, geology or archaeometry, when sectioning or sampling is undesirable. In biology, specimen have a high content of water, which leads to the challenge, that specimen must be fixated to stop a change in elemental distributions due to drying or shrinking during measurements. Most commonly specimens are either freeze-dried or cryo-fixated, the latter being the method of choice regarding the maximal sample integrity. X-ray fluorescence (XRF) techniques like micro-XRF (up to 2D) or confocal micro-XRF (up to 3D) can easily be combined with a cryogenic sample environment, thus, fulfilling the above-mentioned requirements for elemental imaging of biological specimen. In this work, a modified commercial micro-XRF spectrometer (M4 Tornado, Bruker Nano GmbH) is used [1]. The instrument enables micro-XRF as well as confocal micro-XRF with high speed acquisition and lateral resolutions of 30 *m at Cu Kα. We present the addition of a liquid nitrogen Cryo-Jet (Oxford Instruments) which cools the specimen to 120 K rendering longtime measurements of biological specimen feasible. Characterization and stability measurements show the feasibility for cryomeasurements in the laboratory. As a first application, sunflower roots which show different uptake of heavy metals are investigated.

KFM 10.10 Tue 13:00 EMH 225

micro-XRF analysis of color brilliance and dyeing techniques in ancient wool carpet fibers — •Andreas Späth[1], Markus Meyer[1], Thomas Huthwelker[2], Camelia N. Borca[2], Karl Messlinger[3], Manfred Bieber[4], and Rainer H. Fink[1] — [1]1FAU Erlangen-Nuremberg, Physical Chemistry II, Erlangen, Germany — [2]Paul Scherrer Institute, Swiss Light Source, Villigen, Switzerland — [3]FAU Erlangen-Nürnberg, Physiology and Pathophysiology, Erlangen, Germany — [4]Ex Oriente, Waldbrunn, Germany

The vivid and persisting colors of ancient oriental carpets are remarkably stable against any typical form of bleaching. Anthropologists have revived traditional procedures based on fermentation of the wool with G. candidum yeast prior to dyeing. This method results in a higher permeability of the fiber cuticle, but in ancient wool specimens the cuticles are often lost by abrasion and cannot be analyzed anymore. However, the common natural dyes (e.g., alizarin) require the use of a mordant (KAl(SO4)2 or FeSO4) to form a stable mordant-dye-coordination-complex within the keratin fiber. micro-XRF is an excellent tool to detect the distribution of the respective mordant-dye-complexes within the wool [1]. We found that fermentation results in an enhanced concentration of mordant in the inner cortex. Within our recent studies we employ micro-XRF to confirm the fermentation process in specimens from various centuries and cultures - including the oldest knotted carpet known (5th century B.C.) - and gain more insight into the historical spreading of this technique.

[1] M. Meyer et al., Scanning, 2017, 2017, 6346212.

KFM 10.11 Tue 13:20 EMH 225

Applicability of a Laboratory Scan-Free GEXRF Setup for the Investigation of Nano-Layered Samples — •Veronika Szwedowski, Jonas Baumann, Steffen Staeck, Gesa Goetzke, Ioanna Mantouvalou, Daniel Groetzsch, Wolfgang Malzer, and Birgit Kanngiesser — Institut für Optik und Atomare Physik, BLiX, Technische Universität Berlin, Deutschland

Grazing Emission X-Ray Fluorescence (GEXRF) is an analytical method enabling access to information about elemental depth gradients, diffusion or doping profiles of nanoscale samples. A novel scan-free approach significantly simplifies GEXRF setups by using a position and energy dispersive detector, avoiding movable parts in the spectrometer.

The first demonstrations of the technique were shown using synchrotron radiation or laser-produced plasma sources in combination with high-prized detectors enabling fast and stable angle dependent XRF measurements.

In this work a Scan-Free GEXRF (SF-GEXRF) setup with a micro-focus rhodium X-Ray tube and a conventional CCD is introduced which will help to widen the applicability of the technique due to its simplicity and low-cost, while maintaining high efficiency. We show the investigation of two classes of multilayer samples with layer thicknesses in the low-nanometer to sub-nanometer range.