

MM 15: Topical session (Symposium MM): Correlative and in-situ Microscopy in Materials Research

Sessions: Soft Matter and Hybrid Materials; Energy Materials

Time: Tuesday 10:15–13:15

Location: H44

Topical Talk MM 15.1 Tue 10:15 H44
Correlative Microscopy of Biological Cells and Tissues by Scanning X-ray Diffraction, Holography, Tomography and Super-Resolution Optical Microscopy — MARTEN BERNHARDT¹, JAN-DAVID NICOLAS¹, ANDREW WITTMEIER¹, MICHAEL SPRUNG¹, SARAH KÖSTER², and •TIM SALDITT¹ — ¹Georg-August-Universität Göttingen — ²DESY, Hamburg

We present a correlative microscopy approach, which combines holographic x-ray imaging, x-ray scanning diffraction, and super-resolution optical fluorescence microscopy, in particular stimulated emission depletion (STED)-microscopy [1], implemented in the same synchrotron endstation. This allows us to image both labeled and unlabeled molecular components in biophysical and soft matter samples in a quasi-simultaneous scheme. We exploit the complementary contrast mechanisms of x-ray microscopy and optical fluorescence for studies of heart muscle cells: While scanning x-ray diffraction and x-ray holographic imaging provide structural information on the native electron-density distribution within the cell, fluorescent markers visualize specific molecular components.

[1] M. Bernhardt, J.-D. Nicolas, M. Osterhoff, H. Mittelstädt, M. Reuss, B. Harke, A. Wittmeier, M. Sprung, S. Köster, T. Salditt. *Nature Communications* 9: 3641 (2018)

Topical Talk MM 15.2 Tue 10:45 H44
Experimental observations of molecular ordering in slitconfined non-polar fluids — •OLIVER H. SEECK, MILENA LIPPMANN, ANITA EHNES, and FLORIAN BERTRAM — Deutsches Elektronen-Synchrotron DESY, Notkestrasse 85, 22607 Hamburg, Germany

The molecular order of non-polar liquids constrained in slit-geometry of a few molecular diameters exhibits distinct features which cannot be found in bulk liquids. A well-known example is the formation of molecular layering along the surface normal of the confining substrates. Another aspect is the observation that the shear modulus and the viscosity of confined liquids are similar to those observed in the solid bulk phase. The latter phenomenon is known as confinement-induced solidification. Imaging the confined liquid films with Å-resolution in all three dimensions is a great challenge. We have developed a setup for film thicknesses down to 30 Å and which is compatible with X-ray scattering methods such as reflectivity, grazing incidence diffraction and Bragg-scattering. At confined CCl₄-films we have proven the co-existence of a layered phase and a crystalline phase. In the future we will investigate the response of the molecular order to shear forces being applied to the confined films with a new setup.

MM 15.3 Tue 11:15 H44
Establishing a mechanistic model for the synthesis of noble metal nanoparticles — •TOBIAS ZECH¹, TILO SCHMUTZLER¹, DENNIS NOLL¹, KLAUS GÖTZ¹, ERDMANN SPIECKER², and TOBIAS UNRUH¹ — ¹LKS, Friedrich-Alexander-Universität Erlangen-Nürnberg — ²CENEM, Friedrich-Alexander-Universität Erlangen-Nürnberg

In our research we are studying the interaction between gold nanorods (AuNRs) and the stabilizer in form of CTAB, and how the latter influences the morphology of AuNR during the synthesis. AuNRs have the potential to be integral parts in nanoplasmonic devices and in medicine as biomarker and for photothermal therapy. However, lacking reproducibility and low yield make the synthesis expensive. Additionally, CTAB, assumed to strongly adsorb to gold nanoparticles, makes it difficult to functionalize the surface. We use a combination of SANS and SAXS to analyze the behavior of CTAB micelles, gold nanoparticles and their CTAB stabilization layer in-situ. From this we want to develop a mechanistic model, which more accurately describes the anisotropic growth of AuNR. So far, we were able to develop a stabilization model for small gold nanoparticles with CTAB micelles and improve their long-time stability considerably. Furthermore, we could directly correlate the changes of the structure of CTAB micelles to the resulting aspect ratio of AuNRs, when certain additives are present. SAS experiments will play a vital role in developing deeper understanding of the underlying mechanisms of AuNR growth.

15 min. break

Topical Talk MM 15.4 Tue 11:45 H44
In-situ Studies of Electrochemical Interfaces in Lithium-ion Batteries — •HANS-GEORG STEINRÜCK — SSRL Materials Science Division, SLAC National Accelerator Laboratory, Menlo Park, California 94025, USA

In lithium-ion batteries (LIBs), the arrangement of electrolyte molecules directly at the electrode interface, and the electric double layer (EDL) formation are expected to govern the interfacial ion transport during charge/discharge and the electrochemical stability, i.e. the origin and properties of the solid electrolyte interphase (SEI). The SEI is formed on anode surfaces due to electrolyte decomposition at low potentials outside the electrolyte's electrochemical stability window, and dictates cell chemistry, cycle life, and electrochemical reversibility.

Towards this end, the first topic of this talk discusses an Å-resolution combined experimental and theoretical structural determination of solid-liquid interfaces relevant to LIBs, specifically a baseline organic electrolyte containing various concentrations of lithium hexafluorophosphate salt at a metal oxide interface. The second portion of this talk describes a multi-property characterization study of the physical and chemical properties as well as the nucleation and growth of the SEI on silicon anodes relevant to LIBs. We combined in-situ X-ray reflectivity, linear sweep voltammetry, ex-situ X-ray photoelectron spectroscopy, and first principles calculations to develop a holistic understanding of the SEI formation mechanism and properties on native oxide terminated single crystalline Si electrodes.

MM 15.5 Tue 12:15 H44
Nanoscale Characterization of Energy Materials using SIMS based Correlative Microscopy — •SANTHANA ESWARA, ALISA PSHENOVA, JEAN-NICOLAS AUDINOT, and TOM WIRTZ — Advanced Instrumentation for Ion Nano-Analytics, MRT, Luxembourg Institute of Science and Technology, L-4422 Belvaux, Luxembourg

Energy materials play a key role in providing sustainable and eco-friendly energy solutions. To improve the efficiency of energy conversion and storage, modern materials are being increasingly designed at the nanoscale and the chemical compositions are tuned even down to the dopant level. Therefore, nanoscale high-sensitivity chemical analyses are required for (i) establishing the link between local nanoscale characteristics of the material and the overall device performance and (ii) to develop new strategies to enhance performance and improve durability. Our research on this subject is focused on addressing the associated analytical challenges. While Transmission Electron Microscopy (TEM) offers high spatial resolution, the conventional analytical techniques available in TEM are inadequate for the analysis of light elements (H, B, Li) and dopant-level concentrations. Secondary Ion Mass Spectrometry (SIMS) offers high-sensitivity and all elements can be analyzed, but, the lateral resolution is fundamentally limited by ion-solid interaction volume to ~ 10 nm. To overcome this limitation, we use correlative TEM-SIMS [1] to investigate (i) silicon solar cell materials and (ii) electrodes in lithium battery materials. The results, correlative workflow, data treatment methods and potential artefacts will be discussed. [1] L. Yedra et al, *Sci. Rep.* 6, 28705, 2016

MM 15.6 Tue 12:30 H44
Dislocations in Cu(In,Ga)Se₂ solar cell absorbers: Insights from electron microscopy and electronic structure calculations — DANIEL BARRAGAN-YANI¹, EKIN SIMSEK SANLI², and •KARSTEN ALBE¹ — ¹TU Darmstadt, FB 11, FG Materialmodellierung, Otto-Berndt-Str. 3, 64287 Darmstadt — ²Max Planck Institute for Solid State Research, Stuttgart Center for Electron Microscopy, Heisenbergstrasse 1, 70569 Stuttgart

In Cu(In,Ga)Se₂ (CIGSe) based devices, power-conversion efficiencies of more than 15 % can be achieved, although significant dislocation densities are present. This still unexplained behavior is in contrast to what has been reported for other semiconductor materials, where the presence of dislocations strongly affects the electrical properties. This implies that lattice dislocations in CIGSe-based absorbers are per se

electrically inactive or possibly passivated by solute or impurity atoms. In this contribution, calculations based on density functional theory are presented, which reveal the influence of chemical reconstruction and solute segregation on the activity of mixed and partial dislocations in CIGGe. The role of mechanical driving forces for point defect dislocation interaction is highlighted, which allows to explain results obtained from by aberration-corrected scanning transmission electron microscopy in combination with electron energy-loss spectroscopy.

MM 15.7 Tue 12:45 H44

Structural Characterization of the 2D-Powder Material DRCN5T:PC71BM Using X-rays — ●MARVIN BERLINGHOF¹, STEFAN LANGNER¹, CHRISTINA HARREISS¹, ELLA M. SCHMIDT¹, RITA SIRIS², FLORIAN BERTRAM³, JOHANNES WILL¹, STEFANIE RECHBERGER¹, GEORG S. DUESBERG², REINHARD B. NEDER¹, ERDMANN SPIECKER¹, CHRISTOPH J. BRABEC¹, and TOBIAS UNRUH¹ — ¹Friedrich-Alexander-Universität Erlangen-Nürnberg, Erlangen, Deutschland — ²Universität der Bundeswehr München, Neubiberg, Deutschland — ³Deutsches Elektronen-Synchrotron, Hamburg, Deutschland

The crystallinity and with it the power conversion efficiency (PCE) of organic photovoltaic thin films made of small molecule-fullerene blends is tune-able by solvent vapor annealing (SVA). An example of such a system are thin films of DRCN5T:PC71BM. The small molecule DRCN5T crystallizes during annealing with vapors of chloroform, tetrahydrofuran or carbon disulfide. Its crystallites are formed at the surface of the thin film as a 2D powder (oblique crystal system). In the crystallites, DRCN5T assembles itself in stacks, which form the 2D crystalline structure.

A comparison of in- and ex-situ grazing wide angle X-ray scattering (GIWAXS), X-ray pole figures, elastic electron diffraction, and sec-

ondary ion mass spectrometry (SIMS) measurements will be presented. This includes information about, the crystalline structure, preferred orientations, and the vertical composition of the photoactive layer.

MM 15.8 Tue 13:00 H44

Post-treatment processes and their effects on organic solar cell morphology and performance — ●CHRISTINA HARREISS¹, STEFAN LANGNER², STEFANIE RECHBERGER¹, JOHANNES WILL¹, CHRISTOPH J. BRABEC², and ERDMANN SPIECKER¹ — ¹Institute of Micro- and Nanostructure Research & Center for Nanoanalysis and Electron Microscopy, Friedrich-Alexander Universität Erlangen-Nürnberg — ²Institute of Materials for Electronics and Energy Technology, Friedrich-Alexander Universität Erlangen-Nürnberg

During the past decades the efficiency of solution-processed organic solar cells (OSCs) could be improved by developing new processing conditions. Since the morphology of OSCs determines the charge separation at the interfaces and the electron and hole transport to the electrodes, it influences the device performance. Analytical Transmission Electron Microscopy (TEM) techniques like energy filtered TEM (EFTEM) are used to study the morphology since conventional imaging techniques are mostly insufficient to visualize the donor and acceptor. Here we present results on a OSC system composed of a small molecule (DRCN5T) and a fullerene derivative (PC70BM). The active layers were treated by thermal annealing (TA) and solvent vapor annealing (SVA). The SVA was performed with different solvents and for different annealing times. The morphology was revealed for all post-treated active layers by EFTEM and correlated to the performance. Currently we are performing the TA directly in our TEM device by an in situ heating technique. By this, we can observe the phase separation and formation of an interpenetrating network of donor/acceptor phases.