MM 9: Topical session (Symposium MM): Correlative and in-situ Microscopy in Materials Research

Sessions: Advanced Characterization II and III

Time: Monday 15:45–19:00 Location: H44

Topical Talk MM 9.1 Mon 15:45 H44

Scanning transmission electron microscopy as a multidimensional information channel with spatial, momentum and time resolution — •Knut Müller-Caspary[1], Armand Beche[2], Florian Winkler[1], Florian Krause[3], Daen Jannis[2], Andreas Oelsner[4], Heike Soltau[5], Rafal Dunin-Borkowski[1], Sandra Van Aert[2], Johan Verbeeck[2], and Andreas Rosenauer[3] — [1]Forschungszentrum Jülich (D) — [2]EMAT Antwerpen (B) — [3]Universität Bremen (D) — [4]Surface Concept GmbH (D) — [5]PN Detector GmbH (D)

With the advent of ultrafast cameras, methodologies in the field of Scanning Transmission Electron Microscopy experienced a rapid development in the past 5 years. Pixelated STEM now enables the simultaneous acquisition of real and reciprocal space data at unprecedented resolutions and samplings. The methodological framework to measure electric fields at atomic and unit cell scale is presented, based on a robust measurement of the momentum transferred to the STEM probe by the interaction with specimen. We show experimental results on charge density maps in 2D materials and polarisation fields in piezoelectric specimen. Momentum-resolved STEM is additionally discussed as to its potential for the in-situ mapping of electronic properties in electrically biased devices, and the chemical composition mapping via an angular multi-range analysis. Moreover, we report current approaches to enhance the time resolution of ultrafast detectors to the ps-scale, and outline the new opportunities a high sampling of the time domain provides for the characterisation of materials and processes.

MM 9.2 Mon 16:15 H44

Scanning Transmission Electron Beam Induced Current Combined with Spectroscopy for Investigating Energy Conversion in Nanoscale Materials — •Tobias Meyer[1], Birte Kressdorf[2], Jonas Lindner[2], Patrick Peretzki[1], Vladimir Roddatis[2], Christian Jooss[2], and Michael Seibt[1] — [1]4th Institute of Physics: Solids and Nanostructures, University of Goettingen, Friedrich-Hund-Platz 1, 37077 Goettingen, Germany — [2]Institute of Materials Physics, University of Goettingen, Friedrich-Hund-Platz 1, 37077 Goettingen, Germany

Electron Beam Induced Current (EBIC) is a well-established, powerful characterisation tool in semiconductor physics which has been employed in Scanning Electron Microscopes (SEM) for decades, e.g. to map enhanced excess carrier recombination at crystal imperfections. Despite great advancements in instrumentation, the resolution of SEM based setups is rather limited since multiple scattering of the impinging electrons leads to a pear-shaped generation volume and hence a sophisticated crosstalk between primary excitations and the sample's geometry.

Transferring the technique to the Scanning Transmission Electron Microscope (STEM), also referred to as Scanning Transmission EBIC (STEBIC), prevents the electron beam to spread significantly before exiting the sample. Thus, excess carrier dynamics on the nanometer scale become accessible and can additionally be correlated to state of the art STEM signals like Electron Energy Loss Spectra (EELS) and Nano Beam Electron Diffraction (NBED) patterns.

MM 9.3 Mon 16:30 H44

The influence of deformation on the medium-range order of a Zr-based bulk metallic glass characterized by variable resolution fluctuation electron microscopy — •Sven Hilke[1], Harald Rösner[1], David Geissler[2], Annett Gebert[2], Martin Peterlechner[1], and Gerhard Wilde[1] — [1]Institut für Materialphysik, Westfälische Wilhelms-Universität Münster, Wilhelm-Klemm-Str. 10, D-48149 Münster, Germany — [2]Institute for Complex Materials (IKM), Leibniz-Institute for Solid State and Materials Research Dresden (IFW Dresden) Helmholtzstr. 20, D-01069 Dresden, Germany

The medium-range order (MRO) of both as-cast and deformed states of a Zr-based bulk metallic glass (Zr52.5Cu17.9Ni14.6Al10Ti5 (at.-%) - Vitreloy 105) were analyzed using variable resolution fluctuation electron microscopy (VR-FEM). Significant structural changes were observed in the MRO in regions close to and inside shear bands of deformed material, compared with that in the as-cast state (undeformed). As a result of deformation we conclude, that two distinct MRO cluster types were formed in both matrix and shear bands. Moreover, plastic deformation not only alters the MRO in terms of cluster size and volume fraction but also leads to a split into two distinct MRO types. The MRO length scale is larger inside the shear bands compared to the surrounding matrix, which indicates the impact of frictional heating and/or higher atomic mobility. Moreover, these results approve VR-FEM as a potential method for probing deformation stages even within the wide elastic regime of metallic glasses.

MM 9.4 Mon 16:45 H44

Simulation of fluctuation electron microscopy data: the Ka-Xi plot — •Martin Peterlechner, Sven Hilke, and Gerhard Wilde — Institute of Materials Physics, University of Münster, Münster, Germany

The method of fluctuation electron microscopy (FEM), as introduced by Treacy and Gibson, is an excellent tool for analysis of amorphous materials. The signal of the ring intensity variation in a nano-beam diffraction pattern (NBDP) along constant k-values ('Ka' values) is very sensitive to structural changes in the medium range order length scale. To understand the signal and gain knowledge on the sensitivity, we simulated NBDPs by a recently in-house developed multislice algorithm, STEMcl. For every beam position during a scanning transmission electron microscopy (STEM) experiment, the corresponding NBDP was segmented into 150 rings. The 150 corresponding STEM images were calculated, and the profiles of their Fouriertransforms were analysed ('Xi' space). The so obtained 'Ka-Xi' plots are analysed, and conclusions about optimum experimental conditions and the sensitivity of FEM in general are discussed.

MM 9.5 Mon 17:00 H44

Combined in situ heating and diffraction in Scanning Electron Microscopy — •Peter Denninger, Peter Schweizer, Christian Dolle, and Erdmann Spiecker — Friedrich-Alexander University Erlangen-Nuremberg, Cauerstrasse 6, 91058 Erlangen

In this work we introduce a novel heating and diffraction setup in the SEM enabling the simultaneous acquisition of real space and reciprocal space information in situ. Low Energy Nano Diffraction (LEND) in transmission is based on the combination of a fluorescent screen positioned below the sample with a dedicated CMOS camera. The technique has been implemented and successfully tested on graphene and polycrystalline gold. For graphene a hexagonal spot like diffraction pattern can be obtained due to the small convergence angle (hence nano diffraction) typically encountered in SEM. For gold the same is possible for very thin films. With increasing film thickness the contribution of dynamical scattering becomes more prevalent, resulting in familiar transmission Kikuchi diffraction (TKD) patterns. With our setup we could successfully demonstrate LEND down to an energy of 0.5 keV (hence low energy).

A custom-built heating stage for DENS Solution Wildfire NanoChips in combination with the LEND setup offers combined in situ heating, imaging and transmission diffraction in SEM. To showcase the power of this technique the process of solid state dewetting and the aluminum induced Layer Exchange (AlILE) will be shown. In both those processes the combined detection of real- and reciprocal space information lead to new insights about their mechanisms.

15 min. break

Topical Talk MM 9.6 Mon 17:30 H44

Characterization of materials at the nanoscale using hard X-ray microspectroscopy techniques — •Gema Martinez-Criado — ICMM-CSIC, Calle Sor Juana Ines de la Cruz 3, 28049-Cantoblanco, Madrid, Spain

Hard X-ray spectromicroscopy techniques are key tools with relevant applications across multiple fields. In this presentation I briefly describe how these tools are implemented and correlated nowadays for the smart in-situ characterization of advanced heterogeneous materials

at the nanoscale. In addition, the essential role of the associated instrumentation for reliable 2D and 3D data acquisitions with nanometer spatial resolution are shortly presented. Finally, few recent examples under operando conditions exemplify the potential of correlative hard X-ray modalities to provide new insights into nanostructures for novel nanodevices.

MM 9.7 Mon 18:00 H44

Revealing the Influence of Surfactants on the Fast Growth Kinetics of Organic Nanoparticles by In Situ Small-Angle Scattering Studies — •Dennis M. Noll, Isabel Schuldes, Torben Schindler, Tilo Schmutzler, and Tobias Unruh — Institute for Crystallography and Structural Physics, Friedrich-Alexander Universität Erlangen-Nürnberg, Erlangen, Germany

The combination of small-angle X-ray and neutron scattering (SAXS/SANS) with the stopped-flow technique allows for in situ studies of fast structure formation processes. The presented time-resolved experiments enable classical SANS in the regime of few milliseconds for the first time by performing and summing up hundreds of stroboscopic measurements for each sample system.

With in situ SAXS and SANS the fast growth of poorly-water soluble active pharmaceutical ingredient nanoparticles during the antisolvent precipitation process has been studied. To reveal the influence of surfactants on the growth kinetics and the ripening of the organic model system, precipitation with two structurally different and commonly used surfactants, as well as precipitation without any surfactant have been studied. The results of the in situ SANS measurements show an early beginning of ripening already after 250 ms for all sample systems and increased growth kinetics for nanoparticles solubilized by micelles.

Additionally, first in situ SAXS and SANS results of a new and simple model system for small and long-term stable organic nanoparticles with a similar growth behavior will be presented.

MM 9.8 Mon 18:15 H44

In-operando studies on organic field-effect transistors — •Manuel Johnson[1], Tim Hawly[1], and Rainer H. Fink[1,2] — [1]1Lehrstuhl für Physikalische Chemie II, FAU Erlangen-Nürnberg, Germany — [2]CENEM, FAU Erlangen-Nürnberg, Germany

Organic semiconducting films bear high potential for device applications such as field-effect transistors (OFETs). Understanding the correlation between microstructure and charge transport within the active layer of these devices is crucial to achieve high field-effect mobilities. To ad-dress this issue we performed spatially-resolved spectroscopic measurements to correlate the spectroscopic changes with the local microstructure of our thin organic film devices. In our studies we investigated pentacene and different thiophene derivative based OFETs. We could ob-serve small variations in the density of unoccupied states during device operation using X-ray microscopy and local NEXAFS spectroscopy. We attribute the spectral modifications in terms of intramolecular polarization for molecules located at the organic/insulator interface. Spatially-resolved XPS measurements offer additional insight into the absolute change in binding energy (BE). Our recent results contradict the findings from Nagamura et al. that conducted similar studies and reported a BE increase when applying a gate voltage [1]. We discuss the observed decrease in the BE with respect to the operation state of the OFET device and show that it can be used for a visualization of a charge accumulation layer inside the active layer. This research is funded by the DFG within GRK 1896.

[1] N. Nagamura, et al., Appl. Phys. Lett. 106, 2015, 251604

MM 9.9 Mon 18:30 H44

Structural studies on functional and solution-processed organic thin films — •Tim Hawly[1], Manuel Johnson[1], and Rainer Fink[1,2] — [1]Lehrstuhl für Physikalische Chemie II, Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany — [2]CENEM, Friederich-Alexander-Universität Erlangen-Nürnberg, Germany

State-of-the-art organic semiconducting layers offer a variety of advantages compared to conventional inorganic circuitry such as flexibility, low-cost processability and environmental compatibility. However, numerous experiments including vacuum-processed thin films in devices such as organic field-effect transistors (OFETs) suffer from diffusion-limited growth leading to polycrystalline films, which reduce high-mobility charge transport between the source and drain electrode. We utilized spatially resolved XPS to obtain insight into the operating, active layer of a typical OFET. We explored a novel preparation technique utilizing the surface of a liquid (most commonly water) as substrate.[1] Thin-film growth out of solution results in long-range ordered crystalline structures for a variety of small molecules and is ultimately capable of overcoming common domain sizes and grain boundaries present in vacuum-deposited films. Our findings are readily supported by microscopic (AFM, TEM), diffraction (SAED) and spectroscopic (Angle-resolved NEXAFS, STXM) techniques as well as by charge-transport measurements that excel reference data typically by one order in magnitude. This research is funded by the DFG within GRK1896. [1]: C. Xu et al., Angew. Chem. Int. Ed. 2016, 55, 9519-9523.

MM 9.10 Mon 18:45 H44

Structural analysis of complex Liquid Metal Catalyst structures utilizing 3D X-ray and Electron Microscopy — •Janis Wirth[1], Silvan Englisch[1], Christian Wiktor[1], Nicola Taccardi[2], Benjamin Apeleo Zubiri[1], Peter Wasserscheid[2], and Erdmann Spiecker[1] — [1]Institute of Micro- and Nanostructure Research (IMN) & Center for Nanoanalysis and Electron Microscopy (CENEM), Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany — [2]Chair of Chemical Engineering I (Reaction Engineering), Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany

X-ray microscopy (XRM) allows non-destructive 3D investigations of materials across multiple length scales. Due to the high-resolution capabilities and flexible contrast a correlative 3D study combined with scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques is possible. In this contribution, we report about correlative 3D studies of Pd(Pt)-Ga liquid metal catalysts which recently showed outstanding performance in alkane dehydrogenation and, in particular, high resistance against coking. This material exhibits a complex structure featuring a catalytically-active liquid film/droplet layer adsorbed on macroporous silica. 3D characterization across different length scales is required to gain deeper insight into the structure and microscopic mechanisms of the catalyst system. Utilizing XRM the metal droplets and the macroporous silica network can be independently resolved. In order to study the composition of individual metal droplets inside the porous network, site-specific sample preparation has to be combined with analytical TEM techniques.