MM 52: Topical Session: In Situ and Multimodal Microscopy in Materials Physics II

Time: Thursday 10:15–12:45 Location: C 130

Topical Talk MM 52.1 Thu 10:15 C 130

Fast in situ electron tomography: nanoparticles under heat and light excitation — •Wiebke Albrecht — AMOLF, Science Park 104, 1098 XG Amsterdam, The Netherlands

Although standard transmission electron microscopy (TEM) makes it possible to obtain a wealth of information of static (nano)materials, an extension to measurements of dynamic structure-property relationships under realistic conditions is urgently needed. Moreover, with the emergence of nanoparticles with complex shapes, such in situ measurements need to be extended to 3D. Electron tomography (ET) allows for quantifying the 3D morphology of such nanoparticles but is inherently slow. In this talk, I will present how ET can be sped up to be compatible with in situ measurements. In addition, I will show how it can be utilized to quantify heat-induced nanoscale diffusion in single nanoparticles.

MM 52.2 Thu 10:45 C 130

In-situ study of Materials Performance and Structural Properties with high spatial resolution — Anton Davydok and •Christina Krywka — Insititute of Material Physics, Helmholtz-Zentrum Hereon, Hamburg, Germany

Over the past decade, we have seen a rapid development of materials with controlled properties for a wide range of applications from large-scale construction to nanoelectronics. This would not have been possible without a strong contribution from materials science based on experimental studies. The methods and techniques available today allow us to reconstruct the real operation and use of materials with controlled properties and characterize them in real time. In this talk, we will present the applicability and enormous potential of scanning X-ray nanodiffraction for in situ and in-operando studies. The nanofocus end-station of the P03 beamline at PETRA III (DESY, Hamburg), operated by the Helmholtz-Zentrum Hereon, offers unique conditions for in-situ and in-operando experiments in combination with X-ray nanodiffraction. The highly stable experimental setup is designed for structural analyses with submicrometer precision and allows non-destructive access to the interior and properties. The contribution of the P03 Nanofocus Endstation to materials science is demonstrated by the wide range of in-situ experiments already performed at the station, such as mechanical, thermal or electrical tests with organic and non-organic materials. Detailed technical specifications of the beamline and in-situ facilities will be presented as well as the results of the experiments.

MM 52.3 Thu 11:00 C 130

Approaching 1 ns Temporal Resolution in Time-Resolved Electron Holography by Improving Control Signals — •Simon Gaebel, Hüseyin Çelik, Tolga Wagner, Tore Niermann, and Michael Lehmann — Technische Universität Berlin, Germany

Interference Gating (iGate) is a novel and robust method for obtaining time-resolved phase information within transmission electron microscopes (TEM). Its basic idea is a time-dependent suppression of the interference pattern, realized by a deflector in the beam path to which a noise-based control signal is applied. By switching the noise on and off, an interference pattern is generated at short intervals, producing phase information from noise-free periods only.

In this presentation, a novel implementation is presented that aims to achieve increased temporal resolution approaching 1 ns due to an improved control signal. It utilizes the fact that interference patterns not only disappear in the presence of noise, but also when pi-phase shifted patterns are superimposed. The advantage of this approach is the use of square-wave-based control signals (commonly used in telecommunications), which require considerably lower amplitudes and at the same time can be hardware corrected (e.g., impedance matching).

This innovative approach makes iGate interesting for the investigation of processes at higher frequencies, as it enables the recording of phase information in the single-digit nanosecond range and opens new ways of understanding ultrafast phenomena at the nanoscale.

MM 52.4 Thu 11:15 C 130

Interference Gating - A Novel Shutter Mechanism for Time-Resolved Holography — •Tolga Wagner, Hüseyin Çelik, Simon Gaebel, Tore Niermann, and Michael Lehmann — Technische Universität Berlin, Germany

Common shutter mechanisms for realizing time-resolved imaging are usually based either on blocking the intensity of the radiation used (e.g. stroboscopic illumination) or on the fast readout time of modern detectors. For holographic investigations, which are based on the recording and reconstruction of interference patterns, a completely new type of gating mechanism is now available: interference gating (iGate).

The basic idea of iGate is a synchronized destruction of the interference pattern, realized by introducing random phase shifts to the wave, for a defined period of time during a holographic acquisition. The holographic reconstruction process acts as a temporal filter that only retains the information of the undisturbed interferogram outside this period. Since the acquisition of interference patterns is in general very susceptible to external disturbances, a targeted destruction of the interference pattern is rather easy to realize and the interference gating as a method becomes interesting in research areas, in which common shutter mechanisms are difficult to be implemented.

In transmission electron microscopy (TEM), for instance, iGate can be retrofitted with minimal technical effort in almost any existing instrument, hence transforming them into time-resolved investigation techniques at nanosecond timescales with nanometer spatial resolution.

15 min. break

Topical Talk MM 52.5 Thu 11:45 C 130

Probing the Atomic-Scale Internal Phases of Mutliferroic Domain Walls During Dynamics with In-Situ Biasining and Cryogenic STEM — •Michele Conroy — Department of Materials, London Centre for Nanotechnology, Imperial College London, Exhibition Road, London SW7 2AZ, U.K.

Dynamic multiferroic domain wall topologies overturn the classical idea that our nanoelectronics need to consist of fixed components of hardware. To harness the true potential of domain wall-based electronics, we must take a step back from the device design level, and instead re-look at the sub-atomic internal properties. With recent advances in experimental characterisation and theoretical calculation approaches, in the last 5 years reports of non-classic internal structures and functionalities within domain walls have become a common occurrence. As the region of interest is at the nanoscale and dynamic, it is essential for the physical characterisation to be at this scale spatially and time resolved.This presentation focuses on measuring the emergent phases within domain walls during dynamics via in-situ biasing 4D-STEM and EELS. Additionally to explore the low temperature magnetic phases we utilise in-situ cryogenic TEM holders.

MM 52.6 Thu 12:15 C 130

TEM-analysis of electron beam sensitive anodic aluminum oxides at cryogenic temperatures — •Lydia Daum[1], Stefan Ostendorp[1], Martin Peterlechner[1,2], and Gerhard Wilde[1] — [1]University of Münster, Münster, Germany — [2]Laboratory for Electron Microscopy, Karlsruhe, Germany

While the existence of precipitates and segregations with their related stress fields inside of aluminum alloys are essential for strengthening and achieving desired mechanical properties of these, they pose a challenge in the formation of protective anodic aluminum oxide (AAO) coatings on these. In previous studies, those precipitates are classified into three sections based on their performance during anodization. Due to methodological limitations, the experiments were carried out in enlarged intermetallic areas in binary aluminum alloys [1,2]. Here, we employ different pre-treatments to aluminum alloys, in accordance with industrial standards, to address the effect of precipitates on the formation of the electron beam sensitive AAOs. Previous scanning transmission electron microscopy (STEM) studies have shown that cryogenic temperatures can partially reduce the beam damage in materials such as AAOs [3,4]. Here, we apply multimodal electron microscopy to examine both, chemical and structural changes of the precipitates within the AAO and at the interface to the underlying alloy. This work aims for a better comprehension of AAO growth and its corresponding properties. We try to bridge the properties of aluminum alloys gained by precipitations with the optimization of the formation of AAOs, which can offer valuable insights for industrial applications.

MM 52.7 Thu 12:30 C 130

Reducing electron beam-induced defect formation by using in situ TEM gas cell — •Carina B Maliakkal[1], Paolo Dolcet[2], Lukas Braun[2], Maria Casapu[2], Di Wang[1], and Christian Kübel[1] — [1]Institute of Nanotechnology (INT) and Karlsruhe Nano Micro Facility (KNMFi), Campus North, Karlsruhe Institute of Technology (KIT), Germany. — [2]Institute of Technical and Polymer Chemistry, KIT

Ceria – a very common oxide support used in exhaust gas catalysis – was initially investigated to obtain spatially resolved quantitative information about the surface speciation. Electron energy loss spectroscopy (EELS) mapping in a Scanning Transmission Electron Microscope (STEM) was used for this purpose. We did not observe any noticeable change in the crystal structure or surface structure via simple STEM imaging. However, during our investigation to check the Ce oxidation state via EELS, we found that the extent of oxygen vacancies near the ceria surface is strongly affected by the electron beam under the TEM vacuum conditions. This is not surprising, as it is known that the electron beam can significantly alter some materials.[1] Traditional approaches to reduce electron-beam induced damage includes adjusting the high tension, electron dose and dose rate. However, since here the major damage/alteration to the ceria seems to be the induced creation of oxygen vacancies, we demonstrate that by working in oxygen atmosphere, the damage can be successfully compensated for.

Reference [1] Neelisetty et al. Microscopy and Microanalysis 2019 (25) 592.