MM 60: Topical Session: In Situ and Multimodal Microscopy in Materials Physics III

Time: Thursday 15:45–18:00 Location: C 130

MM 60.1 Thu 15:45 C 130

Field ion microscopy contrast in Pt-Ir-Au ternary alloys — •Shalini Bhatt, Shyam Katnagallu, Felipe F Morgado, Baptiste Gault, Christoph Freysoldt, and Jörg Neugebauer — Max-Planck-Institut für Eisenforschung GmbH, Düsseldorf Germany

Field ion microscopy (FIM) allows to resolve the position of atoms in 3D, but the atoms chemical identity can be deduced only indirectly, by local contrast variations. Utilizing our recent EXTRA-FIM approach [1], which adapts the Tersoff-Hamann approach originally developed to Scanning tunneling microscopy (STM) to tunneling-controlled ionization in FIM, we demonstrate a chemical brightness contrast in ternary alloys featuring Pt, Au, and Ir. We correlate the contrast to the electronic structure of the surface, notably the local d-band filling. Yet, the relaxation pattern observed in Au significantly influences the FIM contrast. We, therefore, explore if the contrast information from FIM can be used to correctly interpret the chemical nature and local configurations in FIM experiments. We anticipate that our approach can expand the analytical capabilities of FIM.

[1] Bhatt, S., Katnagallu, S., Neugebauer, J., & Freysoldt, C Phys. Rev. B, 107(23), 235413 (2023)

MM 60.2 Thu 16:00 C 130

Cracking Catalysts: A Synthetic Data Approach to Microscopy Image Segmentation — •Maurits Vuijk, Gianmarco Ducci, Luis Sandoval, Karsten Reuter, Thomas Lunkenbein, and Christoph Scheurer — Fritz-Haber-Institut der MPG, Berlin

In catalysis research, the amount of microscopy data acquired when imaging dynamic processes is typically too vast for non-automated segmentation. The challenge in developing a conventional automated process is that this requires more high-quality annotated training data than available in most cases. In our approach, we thus substitute expert-annotated data with a physics-based synthetic data model.

Our electron microscopy (environmental SEM) data is collected from the process of propanol oxidation to acetone over cobalt oxide. At a certain temperature during the reaction, a phase transition occurs and cracks form on the porous surface, reducing the selectivity of the catalyst. To generate synthetic image data that approximates this transition, our algorithm composes images of the pristine room-temperature catalyst with dynamically evolving synthetic cracks satisfying two physical construction principles. First, crack growth propagates along surface paths which avoid close vicinity to nearby pores. Second, each growing path successively widens and is rendered with increasing contrast to mimic depth over several frames. We then train a neural network model with the sequential data set to obtain a segmented time series of the collected data. This novel method can be used in real-time operation to guide the microscope in capturing the initial nuclei of phase transitions within the system.

MM 60.3 Thu 16:15 C 130

Unusual field evaporation of lithium explained by first principles — •Shyam Katnagallu, Huan Zhao, Se-Ho Kim, Jörg Neugebauer, Baptiste Gault, and Shyam Katnagallu — Max-Planck-Institut für Eisenforschung, Max-Planck-Straße 1, 40237 Düsseldorf, Germany.

Lithium (Li) is a critical metal for battery operation due to its high energy density, low weight, and high mobility. To develop efficient batteries, atomic-scale characterization of complex, Li-containing materials is crucial. Atom probe tomography (APT) could provide extremely valuable insights. However, APT faces many evaporation artefacts that can render the data unusable, particularly for pure Li. We used density functional theory calculations that explicitly include high electric fields to investigate Li's field evaporation behavior from the close-packed Li(110) surface as a prototypical case. At low fields, Li preferentially adsorbs at on-top sites. We systematically study surface-diffusion of Li adatoms as a function of electric field, and discovered the existence of a "critical" electric field below the field strength at which Li evaporates, where the on-top site becomes energetically unfavourable compared to a hollow/bridge site. This leads to a practically barrier-less diffusion of Li atoms on the surface, which explains the spotty evaporation pattern observed experimentally. To prevent this undesired effect, we explored potential approaches to minimize surface diffusion before field evaporation. One approach involves depositing a monolayer of gas such as H, N, or He, onto the Li surface. We show that such an adsorbed gas layer prevents Li atoms from diffusing on the surface.

MM 60.4 Thu 16:30 C 130

Field ion microscopy contrast for Boron in Silicon — •Christoph Freysoldt[1], Shalini Bhatt[1], Jonathan Op de Beeck[2], Claudia Fleischmann[2], and Jörg Neugebauer[1] — [1]Max-Planck-Institut für Eisenforschung GmbH, Max-Planck-Str. 1, 40273 Düsseldorf — [2]Imec, Kapeldreef 75, and KU Leuven, Celestijnenlaan 200D, 3001 Leuven, Belgium

Field ion microscopy (FIM) has been recently shown to improve atom probe tomography (APT), by imaging surface atoms prior to evaporation. This helps to elucidate dynamical surface evolution in APT, and the reconstruction artifacts that may result. In FIM, an imaging gas above the surface is ionized via electron tunneling into empty surface states. The ion flux images individual surface atoms as bright spots. The challenge in FIM is to relate contrast in gas ionization to the relevant atomic surface configurations. We study this here for the case of Boron-doped Silicon, where APT suggests an unintended clustering of the dopants. In FIM experiments with H_2 as imaging gas, a variety of bright features appears upon doping, but the nature of the surface configuration and the source of the contrast is not known. We investigate possible different surface configurations of Boron with density functional theory, as well as their appearance in FIM based on our recent EXTRA-FIM package[1]. We show that electronic structure effects cannot explain the observed bright imaging, and suggest that Boron produces exposed surface clusters during evaporation due to the high strength of Si-B bonds, that hinder homogeneous evaporation.

[1] S. Bhatt *et al.*, Phys. Rev. B **107**, 235413 (2023).

MM 60.5 Thu 16:45 C 130

A Simple and Intuitive Model for Electric Potential Distributions Around TEM-Specimens — •Hüseyin Çelik[1], Robert Fuchs[2], Tolga Wagner[1], and Michael Lehmann[1] — [1]Institute of Optics and Atomic Physics, Technische Universität Berlin, Straße des 17. Juni 135, 10623 Berlin, Germany — [2]Institute of Theoretical Physics, Technische Universität Berlin, Hardenbergstraße 36, 10623 Berlin, Germany

For electron holographic investigations of externally driven real semiconductor nanostructures, a good understanding of the electric potential distribution of the TEM-specimen and the resulting stray fields, especially in electron beam direction, is of great importance. Here, a simple and intuitive model for the approximation of such potential distributions inside and outside semiconductor specimens of a p-n junction, prepared by a focused ion beam (FIB), is presented. The model uses only independent convolutions of the assumed specimen core's potential distribution with a suitable kernel. This allows for the entire potential distribution of a real specimen to be inferred from only one measured projection. Consequently, a significant reduction of the required computational power as well as a drastically simplified measurement process is achieved.

Topical Talk MM 60.6 Thu 17:00 C 130

Spatially and Momentum-Resolved Vibrational Spectroscopy in the Electron Microscope — •Benedikt Haas[1], Peter Rez[2], and Christoph Koch[1] — [1]Department of Physics & Center for the Science of Materials Berlin, Humboldt-Universität zu Berlin, Berlin, Germany — [2]Department of Physics, Arizona State University, Tempe (AZ), USA

Vibrational electron energy-loss spectroscopy was already shown in the 1960s, but it took until 2014 to implement it in an actual electron microscope with sub-nm spatial resolution. This development also allowed for the exploration of larger momentum transfers.

Although optical vibrational spectroscopy techniques (in the form of tip-enhanced methods) have also reached sub-nm resolution, the electron microscope is still unique in being capable of atomically resolving bulk-like specimens, not just surfaces. Recently, we have demonstrated atomically-resolved phonon EELS of extended defects - in excellent agreement with calculations. This study has shown that grain boundaries are not only barriers to phonon transport but can also support localized phonon modes and thus potentially act as phononic waveguides.

Another promising application is momentum-resolved vibrational EELS first demonstrated in 2018. Here, we map phonon dispersion surfaces (in 2D) from momentum-resolved vibrational EELS in combination with suitable theory for quantitative comparison. The technique could be used to visualize anisotropies in phonon transport, e.g. in steady states, or to investigate mode softening.

MM 60.7 Thu 17:30 C 130

Interdiffusion-controlled phase formation at an interconnect interface during soldering — •Sandra Gaertner, Sergiy V. Divinsky, and Gerhard Wilde — Institute of Materials Physics, University of Münster, Münster, Germany

Soldering is a long-standing technique to connect metal parts with a permanent bond. In this process often intermetallic compounds are formed. For the purpose of soldering, elements like Sn, Pb, Bi, Sb, Ag and Cu or their alloys are used in different compositions depending on the respective field of application. Changing environmental regulations towards lead-free solder increased the interest concerning Sn-based solder alloys. Yet, soldering of interconnects involves complex processes related to matter transport, phase stability and phase transformation kinetics. These fundamental processes might depend sensitively on local variations of the microstructure such as microstrain, dislocation accumulation, grain- and phase boundaries or voids. A rigorous analysis of the correlations between the process parameters, the resulting microstructure, the phase formation and the resulting performance necessitates detailed studies on multiple length scales. In the present work, the interdiffusion and diffusion-controlled phase formation processes in a Sn based solder alloy between a Ni based layer and a Cu substrate were investigated by quantitative scanning and transmission electron microscopy. The results are discussed with respect of the fundamental correlations between processing, defect structure and the resulting phase formation sequence.

MM 60.8 Thu 17:45 C 130

Correlative structural and thermal analysis of Zr-based bulk metallic glasses — •Štefan Stanko[1], Jürgen Schawe[1,2], and Jörg Löffler[1] — [1]Laboratory of Metal Physics and Technology, Department of Materials, ETH Zurich, 8093 Zurich, Switzerland — [2]Mettler-Toledo GmbH, Analytical, 8606 Nänikon, Switzerland

Bulk metallic glasses (BMGs) are metallic alloys with a disordered atomic arrangement, obtained via rapid cooling from the melt. Due to their unique properties, BMGs have recently become candidates for additive manufacturing methods such as laser powder-bed fusion (LPBF). In this work, we performed fast differential scanning calorimetry (FDSC) experiments to investigate the crystallization behavior of industrial-grade Zr-based BMGs applied in additive manufacturing. Using a modified FDSC sample holder to allow its integration into a synchrotron X-ray beam, time-temperature-transformation (TTT) diagrams were measured simultaneously with the alloy microstructure. *Ex situ* scanning electron microscopy (SEM) analysis of the samples exposed to the beam revealed that sample degradation during thermal cycling cannot be avoided even when using argon atmosphere. Therefore, TTT diagrams measurements were also performed in a device integrating the FDSC within an SEM, to investigate the degradation *in situ*. The results were correlated with the oxygen content of the alloys to optimize the additive manufacturing process.