MM 6: Topical session (Symposium MM): Correlative and in-situ Microscopy in Materials Research

Advanced Characterization I

Time: Monday 11:45–13:15 Location: H44

Topical Talk MM 6.1 Mon 11:45 H44

Break through new materials characterization frontiers with Atom Probe Microscopy — •François Vurpillot, Benjamin Klaes, Rodrigue Larde, Stefan Parviainen, and Bertrand Radiguet — Normandie Université, UNIROUEN, INSA Rouen, CNRS, Groupe de Physique des Matériaux, 76000 Rouen, France

Atom Probe Microscopy (APM) was proposed in 2012 [1] as a term that embraces the various techniques that derive from field emission generated by tip-like specimen. The most popular and fashion technique of APM is certainly the Atom Probe Tomography (APT) as evidenced by the dramatic increase in the number of books and publications in this field [1-4]. If now APT has its place among the panel of microscopy techniques, this instrument reaches its frontiers due in part to instrumental limitations, but more fundamentally intrinsic physical limitations. Break through these frontiers is however possible by correlating experimental data with accurate modelling and simulation approaches. This presentation will focus on different examples showing the capabilities of APM to image atomic defects in 3D and in the real space. The impact of the stress induced by the high field application is discussed from the quantum scale to the mesoscopic scale.

[1]*B. Gault et al., Atom Probe Microscopy, Springer, 2012. [2]*Lefebvre et al., Atom Probe Tomography Put Theory Into Practice, Academic Press, 2016. ; Larson et al.., Local Electrode Atom Probe Tomography, A User's Guide, Springer, 2013 ; Miller and Forbes, Atom-Probe Tomography, The Local Electrode Atom Probe, Springer, 2014

MM 6.2 Mon 12:15 H44

Determination of 3D electrostatic field at an electron nano-emitter — •Mingjian Wu[1], Alexander Tafel[2], Peter Hommelhoff[2], and Erdmann Spiecker[1] — [1]Institute of Micro- and Nanostructure Research & Center for Nanoanalysis and Electron Microscopy (CENEM), Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), 91058 Erlangen — [2]Department Physik, Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), 91058 Erlangen

Revealing and quantifying the 3D electrostatic field of field emission nano-emitters is key to answer the fundamental question of how the field interacts with the sharp tips. Here, we determine the 3D electrostatic field in situ at an electron nano-emitter. Differential phase contrast in scanning transmission electron microscopy has been applied to image nanoscale electrostatic fields of a sharp tungsten electron emitter with an apex radius of about 20 nm and under field emission condition. Assuming axial symmetry of the nano-emitter, we derived a method based on the inverse Abel transform to quantitatively reconstruct an axial slice of the 3D electrostatic field from a single projection measurement. The highest field strength of 2.92 V/nm is measured at the nano-emitter apex under condition of a bias voltage of -140 V with respect to the grounded counter electrode located about 650 nm from the apex, resulting in an emission current of more than 2 uA. The experimental results are compared with simulations based on a finite element numerical Maxwell equation solver. Quantitative agreement between experiment and simulation has been achieved.

MM 6.3 Mon 12:30 H44

Cryo-atom probe tomography for in-situ diffusion measurement of H at crystal defects — •Peter Felfer — Institute for General Materials Properties, Department of Materials Science, Friedrich-Alexander Universität Erlangen-Nürnberg

The diffusion of H in metallic materials is a highly important phenomenon in for both hydrogen energy conversion and structural integrity of materials. Historically, diffusion experiments at the crystal defect scale have not been possible due to measurement instrumentation limitations. As a result, the theoretical description of the phenomenon lacks experimental data to be compared to on individual defects. In recent years atom probe tomography has shown to be capable of delivering such data. About a decade ago atom probe has first been used to locate H, more specifically D in metals. Initially, deuteration through gas charging was used to create D dissolution in thermodynamic equilibrium, which was then supplemented by electrochemical charging, which can be used to create supersaturated states. Since for Fe, the most common base metal in engineering in the form of steel, has very low residual solubility for H even at room temperature, but still a high diffusion coefficient, cryo-cooling can be used to *freeze* D in the material and trigger diffusion only at controlled time intervals. In this talk, I will present first results of such a diffusion experiment. I will also present the design and first results of direct H (no D) measurements in metallic materials using a new atom probe with very low H background.

MM 6.4 Mon 12:45 H44

Atom Probe Tomography for Thermoelectric Materials — •Yuan Yu[1], Oana Cojocaru-Mirédin[1], and Matthias Wuttig[1,2] — [1]I. Physikalisches Institut (IA), RWTH Aachen Univeristy, Sommerfeldstraße, 52074 Aachen, Germany — [2]JARA-Institut Green IT, JARA-FIT, Forschungszentrum Jülich GmbH and RWTH Aachen University, 52056 Aachen, Germany

Both microstructures and chemical composition of defects, including 0D point defects, 1D dislocations, 2D interfaces, and 3D precipitates, significantly influence the electrical and thermal transport properties of thermoelectric materials. However, determining their three-dimensional chemical composition, with a spatial accuracy of sub-nanometer and chemical sensitivity of ~10 ppm was impossible until the emergence of atom probe tomography (APT). In this work, we summarize the existing studies on local composition of various structural defects in thermoelectric materials using APT. With the chemical information at sub-nanometer scale, the corresponding thermoelectric properties can be better understood. Moreover, APT can distinguish a peculiar bonding mechanism, which is called metavalent bonding being responsible for intrinsic low lattice thermal conductivity, between matrix and defects. This provides a novel method to analyze and adjust the tradeoff between intrinsic bonding and extrinsic defects. Thereby, this precious technique is able to extend the concept of defect engineering and adjust the balance between bonding and defects in thermoelectrics and facilitate the rational design of high-performance thermoelectric materials.

MM 6.5 Mon 13:00 H44

Magic Colloidal Clusters: 3D Investigation of Self-assembled Polystyrene Particles forming Agglomerations in Crystalline Order using X-Ray Nano- and Electron Tomography — •Silvan Englisch[1], Janis Wirth[1], Thomas Pryzbilla[1], Benjamin Apeleo Zubiri[1], Junwei Wang[2], Nicolas Vogel[2], and Erdmann Spiecker[1] — [1]Institute of Micro- and Nanostructure Research , Friedrich Alexander-University of Erlangen-Nuremberg (FAU), Germany — [2]Institute of Particle Technology at the FAU

X-ray tomography (XRT) and electron tomography (ET) allow 3D investigations across multiple length scales. Morphological segmentation of the tomographic reconstructions of complex sample structures enables in-depth quantitative and position-correlating analyses. So-called magic colloidal clusters are self-organized structures (or assembies) of polystyrene colloidal particles. XRT/ET were employed to study the exact 3D positions of each individual primary particle. A sufficiently high contrast in the acquired reconstructions, which is important for a reliable segmentation of the 3D datasets, is challenging to achieve since the samples are reaching the limits of XRT (resolution) and ET (size). On the one side, a morphology-threshold filter (software: Arivis) was applied to segment the original reconstructions and determine the 3D positions of the spherical and monodisperse primary particles. On the other side, spheres with a particle-size matching diameter were fitted to these positions in a virtual volume. These workflows allow to investigate the exact structure and possible defects in such magic colloidal clusters.