MM 52: Topical session: In-situ Microscopy with Electrons, X-Rays and Scanning Probes in Materials Science VI - Structural transitions

Time: Thursday 11:45–13:15 Location: H38

15 min. coffee break

MM 52.1 Thu 12:00 H38

Grain growth of nc-Pd during heating using ACOM-STEM — •KK Neelisetty[1,4], A Kobler[1,4], VSK Chakravadhanula[1,3,4], T Scherer[1,2], H Hahn[1,3,4], and C Kübel[1,2,3] — [1]INT, Karlsruhe Institute of Technology (KIT), Germany — [2]KNMF, KIT, Germany — [3]HIU, KIT, Germany — [4]TU Darmstadt, Germany

Understanding grain growth in nanocrystalline (nc) metals and how it can be controlled and tuned is a major objective for applications and provides new fundamental insights. In-situ heating experiments in TEM using Automated Crystal Orientation Mapping (ACOM) is a powerful approach to reveal the microstructure and orientation of grains in nanoscale and follow it's structural evolution. In this study, sputtered ncPd thin-films about 50 nm thick were used to evaluate the grain growth in 2D. Mapping grain orientation in an area initially containing more than thousand grains with a resolution of 1.5 nm allowed for a statistically meaningful analysis of grain orientation, presence of twins, grain rotation and grain boundary migration over time at elevated temperature. Evaluation of maps was performed using MATLAB using Mtex tool-box, providing information about the local misorientations between grains, CSL boundary, twin density and grain size distribution, texture, and number of neighbors of selected growing grains. Global analysis of grains growing is in good agreement with the structural changes observed in bulk samples. Local analysis shows that grain boundary curvature and grain boundary angle are not sufficient criteria to explain grain growth in nc-metals, but triple- and quadruple line networks appear to stabilize nanograins locally.

MM 52.2 Thu 12:15 H38

Solid-state Dewetting of Metallic Thin Films Studied by Complementary in situ Transmission Electron Microscopy Techniques — •Florian Niekiel, Simon M. Kraschewski, Peter Schweizer, Benjamin Butz, and Erdmann Spiecker — Institute of Micro- and Nanostructure Research & Center for Nanoanalysis and Electron Microscopy (CENEM), Universität Erlangen-Nürnberg, Erlangen, Germany

The transition of a thin film into an energetically favorable set of particles at temperatures below the melting temperature of the bulk material is denominated as solid-state dewetting. This phenomenon is on the one hand an undesirable degradation and failure mechanism in thin film applications. On the other hand it can deliberately be exploited to generate nanoparticle arrays with controlled shape, ordering and composition.

In this work we use complementary in situ transmission electron microscopy (TEM) techniques with a chip-based heating system to study the underlying physics during solid-state dewetting. The techniques employed range from electron diffraction over scanning transmission electron microscopy (STEM) to high-resolution TEM. The combination of statistical information with microscopical information down to the atomic scale provides insights into the kinetics, texture evolution and mechanisms involved.

MM 52.3 Thu 12:30 H38

Suppression of phase transformations in Nb-H thin films — •Vladimir Burlaka, Stefan Wagner, and Astrid Pundt — Universität Göttingen, Institut für Materialphysik, Friedrich-Hund-Platz 1, 37077 Göttingen

Mechanical stress in the GPa-range is known to arise during hydrogen absorption in metal films adhered to rigid substrates.[1] It can be released by plastic deformation. For thin films, stress release is much less efficient than for thicker films: It can be even suppressed, and very thin films can stay in a highly stressed state.[2] This influences the thermodynamic and kinetics properties of the system, and may suppress phase transformations.[3] In this presentation, we use Nb-H films as a model system to study the presence of phase transformations depending on the film thickness (d $<$ 110 nm). Phase transformations can be monitored by studying surface corrugations with the scanning tunneling microscope (STM).[4] As soon as hydride precipitates form inside the metal film, a local height change will be detected in the film surface topography. We have demonstrated that this method is more sensitive than conventional XRD.[5] STM allows for finding a Nb-film thickness, below which no sign of a phase transformation can be found at 300K. Complementary methods support this experimental result. This research is kindly supported by the DFG via the projects PU131/12-1 and PU131/9-2. [1] A. Pundt et al. Annu. Rev. Mater. Res. 36 (2006) 555. [2] M. Hamm et al., APL 106 (2015) 243108. [3] S. Wagner et al., Int.J.Hydr.En., in print. [4] K. Nörthemann et. al., PRB 78 (2008) 014105. [5] V. Burlaka et al., JALCOM 645 (2015) 388.

MM 52.4 Thu 12:45 H38

Influence of patterned stress states on hydrogen loading in Vanadium thin films studied by electrochemical hydrogenography — •Anshu Tyagi, Florian Döring, Hans-Ulrich Krebs, and Astrid Pundt — Universität Göttingen, Institut für Materialphysik, Friedrich-Hund-Platz 1, 37077 Göttingen, Germany

It has been shown that the mechanical stress state of a metal film sensitively affects the chemical potential of hydrogen in the metal.[1] The stress state of a film can be changed by the adhesion of the metal film to the substrate and can be modulate by patterning the adhesion conditions. [2] Stress sensitive Vanadium thin films act as model systems in this work. These films are sputter deposited onto transparent glass substrates. Patterning is performed using Palladium and Polycarbonate layers. The hydrogen uptake in these films is studied using Hydrogenography which monitors the film transparency and reflectivity.[3] We apply this method using electrochemical loading in a light-microscope. This electrochemical Hydrogenography allows to optically probe for the local hydrogen concentration.

Comparative studies on different adhesion conditions are done by in-situ EMF measurements. This provides the related chemical potentials including information about the phase boundaries. The transmission and reflection spectra are discussed with respect to these data.

References [1] S.Wagner, A. Pundt, APL 92 (2008) 051914. [2] A. Pundt, E. Nikitin, R. Kirchheim, P. Pekarski, Acta Mater. 52 (2004) 1579. [3] R. Gremaud. M. Slaman, H.Schreuders, B.Dam, and R.Griessen, APL 91 (2007) 231916.

MM 52.5 Thu 13:00 H38

Transmission electron microscopy image and energy-dispersive X-ray map simulations of Ga-covered Pb nanoparticles embedded in an Al matrix. — •Martin Peterlechner and Gerhard Wilde — Institut für Materialphysik, WWU Münster, Münster, Deutschland

Nanoparticles are in focus of research since decades due to their size effects. Embedded nanoparticles in a matrix show additional effects caused by their interface structure. In the present study, the simulation of transmission electron microscopy (TEM) images is focused. High-resolution TEM (HRTEM) contrast, Scanning-TEM (STEM) contrast and energy-dispersive X-ray (EDX) maps are elucidated. A simple atomistic model of an Al matrix with Ga-covered embedded Pb nanoparticles was chosen to study moiré contributions and thickness effects for contrast formation. HRTEM images of the as-generated structures were simulated using the multislice algorithm by Kirkland, and STEM/EDX micrographs were simulated using muSTEM by D'Alfonso, Findlay and Allen. The spacing of the moiré pattern can be used to determine the difference of the involved lattice planes with a high accuracy, however, measurements of absolute atomic positions are not always possible since atoms can become 'invisible' at local regions of the moiré pattern. Moreover, apparent interface roughening can occur by moiré effects. The EDX signal of the Ga atoms is blurred with increasing thickness. Therefore, image simulation is inevitable to interpret atomic positions and chemistry at interfaces, in particular when a nanoparticle is embedded and not of constant thickness.