MM 24: Topical session (Symposium MM): Correlative and in-situ Microscopy in Materials Research

Sessions: Energy Materials II; Nanomechanical Testing

Time: Wednesday 10:15–13:15 Location: H45

Topical Talk MM 24.1 Wed 10:15 H45

Correlating electrical and mechanical behaviour of polymer supported metal thins with in-situ methods — •Megan J. Cordill — Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria

Ductile films and lines are an integral part of flexible electronics because they allow current flow between semiconducting islands and other operating features. However, the charge carrying ductile films must be of an optimum thickness and microstructure for suppression of cracking when tensile loading is applied. Studies of strained films on polymer substrates tend to emphasize only the electrical properties and thickness effects more than the role of film microstructure or deformation behavior. To address both the electro-mechanical and deformation behavior of metal films supported by polymer substrates, in-situ resistance measurements were performed with in-situ confocal scanning laser microscopy imaging of the film surface during cycling. The resistance measurements allow for the examination of the changes in resistance with strain, while the surface imaging permits the visualization of localized thinning and crack formation. Furthermore in-situ synchrotron tensile tests provide information about the stresses in the film and show the yield stress where the deformation initiates and the relaxation of the film during imaging. The combination of electrical measurements, surface imaging, and stress measurements allow for a complete picture of electro-mechanical behavior needed for the improvement and future success of flexible electronic devices.

MM 24.2 Wed 10:45 H45

Correlation of conductivity and failure mechanism of silver nanowire networks: a scale bridging in situ study — •Nadine Schrenker[1], Peter Schweizer[1], Marco Moninger[1], George D. Spyropoulos[1], Mirza Mačković[1], Manuela Göbelt[2], Nikolas Karpstein[1], Silke Christiansen[2], Christoph J. Brabec[1], and Erdmann Spiecker[1] — [1]FAU Erlangen, Germany — [2]MPI, Erlangen

For flexible organic solar cells it is decisive to withstand mechanical loading without sacrificing the performance. Silver nanowire (Ag NW) networks are a highly promising electrode material, since they combine a low sheet resistance with a high transmittance, however the interplay between microscopic failure mechanisms and the functional properties of electrodes in complete OSCs has not been elucidated so far. In this work we introduce a scale-bridging in situ approach to correlate the mechanical response of Ag NW electrodes with their electrical properties starting from single wires up to complete solar cell devices. On the nanometer scale in situ STEM tensile tests of single 5-fold twinned Ag NWs reveal a localized deformation by necking and a clear size-dependence of the mechanical properties. Going up in scale, Ag NW networks on PET foils were tested by in operado SEM tensile tests. These tests show a clear dependency of the wire orientation regarding the straining direction. Buckling as well as kinking as deformation mechanism were observed for wires perpendicular to the straining direction. Moreover, the texture can be utilized to increase the conductivity at 20 % up to nine times. The phenome of kinking is analyzed via HRTEM and complementary atomistic simulation.

MM 24.3 Wed 11:00 H45

In Situ and Ex Situ Electron Microscopy Studies of Al-Si Alloying at Grain Boundaries — •Christoph Flathmann[1], Hendrik Spende[2], Tobias Meyer[1], Patrick Peretzki[1], 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 Semiconductor Technology, Braunschweig University of Technology, Hans-Sommer-Straße 66, 38106 Braunschweig, Germany

During alloying of thin aluminium (Al) layers with bulk multicrystalline (mc) silicon (Si), Al entirely melts at temperatures above 660 °C whilst Si remains solid. Since a surplus of Si is available, the equilibrium concentration of the melt is given by the silicon-rich liquidus line at the corresponding temperature. Thus, a thermodynamic driving force for silicon dissolution is present. However, for mc Si, grain boundaries are sites of preferred dissolution. Hence, uneven dissolution is expected in mc material.

Employing scanning electron microscopy (SEM) and electron beam induced current (EBIC), preferential dissolution, at both low symmetry grain boundaries and {111} twin boundaries, is observed for ex situ alloyed samples. Correlating SEM and EBIC signals allows for visualising the dependence of dissolution depth on electrical junction formation. Furthermore, the dynamics of preferential dissolution are studied by in situ TEM heating experiments of {111} twin boundaries.

MM 24.4 Wed 11:15 H45

3D characterization of macroporous MFI-type zeolite crystals combining nano X-ray tomography and 360° electron tomography — •Dominik Drobek[1], Janis Wirth[1], Silvan Englisch[1], Tobias Weissenberger[2], Benjamin Apeleo Zubiri[1], Wilhelm Schwieger[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

Combining lab-based high-resolution X-ray microscopy (XRM) tomography with 360° electron tomography (ET) enables three-dimensional (3D) imaging of complex material structures across multiple length scales. MFI-type zeolite crystals, which are synthesized utilizing steam-assisted crystallization with mesoporous silica spheres as template[1], with sizes of around 3 μm featuring a well-connected intracrystalline macropore network approach the upper (XRM) and lower (ET) resolution limits of each technique. The combination of both techniques enables a complete 3D reconstruction of the pore structure enabling for quantitative analyses and, furthermore, a direct comparison of the tomography techniques themselves. For optimized imaging conditions individual particles are prepared on a tip utilizing the stamping transfer technique[2] in a dual-beam SEM/FIB instrument. References: [1] A.G. Machoke et al., Adv. Mater. 27 (6) 1066-1070, 2015. [2] T. Przybilla et al., Small Methods 2, 1700276, 2018.

15 min. break

Topical Talk MM 24.5 Wed 11:45 H45

Pathing the way to unique, nondestructive 3D-microstructure properties by in situ Laue tomography — Jean-Baptiste Molin[1], Loic Renversade[2,3], Jean-Sebastien Micha[2,3], and •Christoph Kirchlechner[1] — [1]Max-Planck-Institut für Eisenforschung GmbH — [2]Univ. Grenoble Alpes, CNRS, CEA, INAC-SyMMES, — [3]CRG-IF BM32, ESRF, BP 220

Interlinking the mechanical properties of materials to their underlying and evolving microstructure is vital for a mechanism-based understanding of deformation, in particular at the micron scale. Numerous characterization tools, like scanning electron microscopy (SEM), transmission electron microscopy (TEM) as well as synchrotron based Laue microdiffraction (Laue) offer a complementary toolbox being well suited to answer most questions in material science today.

However, the current toolbox is either limited to surface information (e.g. SEM), thin samples (TEM) or integrated information (Laue). To close this gap we have combined a DAXM setup with a nanoindenter at BM32 of the European Synchrotron (ESRF). The talk will primarily focus on the experimental aspects of this unique machine including its experimental limits as well as prospects of future use.

MM 24.6 Wed 12:15 H45

Local Fatigue Behavior of Bimodal Copper Sheets Investigated by Dynamic Micropillar Compression — •Sebastian Krauss, Benoit Merle, and Mathias Göken — Friedrich-Alexander-Universität Erlangen-Nürnberg, Lehrstuhl Allgemeine Werkstoffeigenschaften (WW I), Erlangen, Germany

In contrast to macroscopic fatigue testing, fatigue experiments on local microsamples offer the opportunity to isolate the individual microstructural contributions from the global behavior of layered materials. In this study, Accumulative Roll Bonding (ARB) processed copper sheets with a bimodal microstructure were analyzed. Micropillars were fabricated by Focused Ion Beam (FIB) milling, yielding testing specimens positioned inside the individual layers of the material. Due to the bimodal microstructure, the microsamples from the different layers show varying grain sizes, which results in a change of the respective fatigue properties. Additionally, micropillars were prepared at the interface to study interface contributions to the fatigue behavior. The investigations were executed by a novel approach that combines dynamic nanoindentation and micropillar compression. With this technique the high cycle fatigue range is easily accessible for microscale samples. Observation of the underlying deformation processes was performed by SEM imaging as well as FIB cross-sectioning of the deformed samples.

MM 24.7 Wed 12:30 H45

Mechanical properties of nanoporous gold studied by scale-bridging in situ testing, non-destructive 3D analyses and experimentally-informed simulations — •Thomas Przybilla[1], Erich Thiess[1], Florian Niekiel[1], Benjamin Apeleo Zubiri[1], Mirza Mačković[1], Peter Schweizer[1], Zhuocheng Xie[2], Julien Guénolé[2], Arun Prakash[2], Stephen T. Kelly[3], Hrishikesh A. Bale[3], Erik Bitzek[2], and Erdmann Spiecker[1] — [1]IMN, FAU, Erlangen, Germany — [2]WW 1, FAU, Erlangen, Germany — [3]Carl Zeiss X-ray Microscopy, Pleasanton, USA

The objective of this study is to understand the interplay between the size of single struts, the network topology/morphology and the defect structure in nanoporous gold (npg). The aim is realized by combining in situ micromechanical testing with non-destructive tomographic techniques and experimentally-informed simulations. Micropillar compression is performed in scanning electron microscopy and transmission electron microscopy. For small strut sizes, 360° electron tomography is applied enabling high quality reconstructions of the npg network. The experimentally derived 3D data are used as input for large-scale atomistic simulations and compared with simulations on geometrically constructed structures. This approach allows the correlation of experimental and simulated flow stress, the explanation of defect mechanisms observed in the experiment and the study of different topology and boundary conditions. For larger strut sizes analyses are carried out by high-resolution X-ray tomography and experimentally-informed finite element simulations correspondingly.

MM 24.8 Wed 12:45 H45

Experimentally-Informed Large-Scale Atomistic Simulations of Nanoporous Gold — •Zhuocheng Xie[1], Julien Guénolé[1], Aruna Prakash[1], Thomas Przybilla[2], Erdmann Spiecker[2], and Erik Bitzek[1] — [1]Materials Science & Engineering, Institute I, Friedrich-Alexander-Universität Erlangen-Nürnberg, Martensstraße 5, 91058 Erlangen, Germany — [2]Institute of Micro- and Nanostructure Research & Center for Nanoanalysis and Electron Microscopy (CENEM), Friedrich-Alexander-Universität Erlangen-Nürnberg, Cauerstraße 6, 91058 Erlangen, Germany

Nanoporous gold (NPG) is an ideal model system for the study of size effects at the nanoscale, since the ligament size can be precisely tailored within the nanometer to micrometer range. In this work, we study the deformation behavior of NPG using large-scale molecular dynamics simulations. We report on uniaxial compression tests using experimentally informed real-size atomistic samples, which are reconstructed from electron tomography datasets. By comparing the results of these simulations with simulations on geometrically constructed samples with average ligament size and porosity equal to the experimental samples, we study the influence of morphology and topology on the deformation behavior of NPG. The influence of surface-induced-stress on the critical resolved shear stress of NPG is evaluated by comparing the real-size samples and samples scaled-down by different factors. Additionally, the deformation mechanisms are studied in detail. The results provide qualitative insights in the fundamental deformation mechanisms not readily observable in in-situ mechanical tests.

MM 24.9 Wed 13:00 H45

Fracture toughness of predeformed chromium studied with microcantilever bending — •Stefan Gabel, Benoit Merle, Erik Bitzek, and Mathias Göken — Institute I: General Materials Properties, FAU Erlangen-Nürnberg, Germany

Cr, Mo and W are the most common bcc metals, which are characterized by a high melting point and high strength. However their fracture toughness at room temperature is low. This is due to their rather high ductile by brittle transition temperature. At room temperature the fracture toughness is limited by dislocation mobility or by the inability to activate nucleation sources. While this behavior is well researched for W, there are only few studies for Cr. FIB milled cantilevers were used to characterize the facture toughness of Cr on the microscale and to study the influence of the loading rate and the initial dislocation density of the sample. In order to achieve high dislocation densities by pre-deformation, Vickers indentations were made on the sample surface prior to testing. The resulting plastic strain field in the material was analyzed by FEM. Selecting different distances to the center of the Vickers indent allows varying the amount of pre-deformation of the cantilevers. The measurements showed that an increase of the loading rate embrittles Cr, whereas an increase of the dislocation density leads to a toughening of the sample. Furthermore the dependence of the transition in the fracture behavior on the availability dislocations and activity of dislocations sources was investigated via TEM-lamella lift-outs and Transmission Kikuchi Diffraction mapping.