# MM 28: Topical session (Symposium MM): Correlative and in-situ Microscopy in Materials Research

Sessions: Complex and Disordered Materials I and II

Time: Wednesday 15:00–17:45

### Topical Talk

## MM 28.1 Wed 15:00 H45 Towards understanding dislocation based plasticity in high entropy alloys by in-situ TEM — •GERHARD DEHM — Max-Planck-Institut für Eisenforschung GmbH

Dislocation-based deformation mechanisms of high entropy alloys (HEAs) remain elusive and require a fundamental understanding in order to tailor their mechanical properties. Since HEAs have five or more constituent elements close to the equiatomic ratio with a stable single phase, their plastic deformation is expected to be different compared to conventional alloys. Here, we present a study correlating the microstructure and dislocation plasticity of a single crystalline FeCrCoMnNi FCC single phase HEA by employing in-situ transmission electron microscope (TEM) compression and tensile deformation. Moreover, an atomic-scale chemical analysis is conducted by aberration-corrected scanning TEM energy dispersive X-ray spectroscopy (STEM-EDS) and atom probe tomography to investigate chemical inhomogeneity, for example, precipitate formation or local inhomogeneity. The aims of the study are understanding of dislocation plasticity in a FCC HEA, investigation of nanometer-scale elemental distribution and measurement of mechanical properties of FCC HEA submicron pillars. Acknowledgements: Major contributions by S. Lee, W. Lu, C. Kirchlechner, R. Raghavan, Z. Li, J. Duarte, S.H. Oh, D. Raabe and C. Liebscher are gratefully acknowledged.

MM 28.2 Wed 15:30 H45 Electron beam-induced rejuvenation in amorphous TiAl during in-situ TEM deformation — Christian Ebner<sup>1</sup>, Ja-GANNATHAN RAJAGOPALAN<sup>2</sup>, CHRISTINA LEKKA<sup>3</sup>, and •CHRISTIAN RENTENBERGER<sup>1</sup> — <sup>1</sup>University of Vienna, Physics of Nanostructured Materials, Vienna, Austria-  ${\rm ^2}Arizona$  State University, Department of Materials Science and Engineering, Tempe, USA — <sup>3</sup>University of Ioannina, Department of Materials Science and Engineering, Ioannina, Greece

In-situ transmission electron microscopy (TEM) facilitates the combined study of structure and fundamental mechanical properties at the nanometer scale. With the advancement in tools, methods and evaluation the sensitivity of measurements became increased. We show that electron irradiation of amorphous TiAl thin films under external tensile stress results in structural rejuvenation and a characteristic change of the elastic properties over time as measured by the atomic-level elastic strain contained in the TEM diffraction pattern. Deeper insights into the property changes triggered by the electron beam and resulting in the observed material response are gained via classical molecular dynamics simulations. These simulations reveal a beam-induced change in quantities like mean atomic volume, potential energy and atomic vibrational mean square displacement. The direct link to the experimental data is established by the calculation of diffraction pattern based on the simulated atomic structure.

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# MM 28.3 Wed 15:45 H45

Micro & Nanomechanics of the Superplastic Alloy Zn-22% Al - Effect of Sample Size on Ductility - • PATRICK FELD-NER, MATHIAS GÖKEN, and BENOIT MERLE — Materials Science & Engineering, Institute I, Friedrich-Alexander-University Erlangen-Nuremberg, 91058 Erlangen, Germany

Superplastic micro & nanoforming has a great potential for a high throughput production of small-scale structural devices with complex geometries. However, it has not yet been established if the macroscopically observed superplastic behavior also persists at microscopic length scales and which fundamental processes govern structural superplasticity in metallic alloys. For this reason, the micro & nanomechanical properties of the superplastic alloy Zn-22% Al were characterized as a function of the specimen size, using nanoindentation at elevated temperatures, in situ pillar compression, as well as in situ tensile testing in a TEM. The measured local strain-rate sensitivity and apparent activation energy provided a strong evidence for superplastic like flow at the micrometer-scale. However, below a critical specimen volume Location: H45

a breakdown of the superplastic flow behavior is revealed, which is accompanied by a change of the apparent rate dependency. Based on the failure morphologies observed during in situ testing, this change of the rate-controlling deformation process is discussed in terms of a transition from boundary mediated ductility to boundary mediated brittleness.

MM 28.4 Wed 16:00 H45 Plasmon characteristics of shear bands in an Al88Y7Fe5 metallic glass — •MAXIMILIAN GROVE<sup>1</sup>, MARTIN PETERLECHNER<sup>1</sup>, HARALD RÖSNER<sup>1</sup>, GERHARD WILDE<sup>1</sup>, ROBERT IMLAU<sup>2</sup>, and ALESSIO  ${\tt Zaccone^3}$  —  ${\tt ^1Institute}$ für Materialphysik, Westfälische Wilhelms-Universität Münster, Wilhelm-Klemm-Str. 10, D-48149 Münster, Germany — <sup>2</sup>FEI Company, Achtseweg Noord 5, 5600 KA Eindhoven, The Netherlands — <sup>3</sup>Department of Physics, University of Milan, Via Giovanni Celoria, 16, 20133 Milan, Italy

Shear bands were observed as a result of plastic deformation in coldrolled Al88Y7Fe5 metallic glass. They display alternating density changes with respect to the matrix (high and low density segments) along their propagation direction. Electron Energy loss spectroscopy (EELS) was used to map the volume Plasmons of shear bands and surrounding matrix. A characteristic Plasmon energy shift of about 10-20 meV relative to the matrix was found for both high and low density segments. For precise analysis, an open source python module (Hyperspy) was utilized to fit the peak shapes by Lorentzian functions and to visualize e.g. the Plasmon energy shift, peak width and height. The experimental results are discussed with respect to the ionic density and the plasmon-phonon interaction.

### 30 min. break

**Topical Talk** MM 28.5 Wed 16:45 H45 **Recent advances in in situ TEM** — •CHRISTIAN KÜBEL<sup>1</sup>, C.N. SHYAM KUMAR<sup>1,2</sup>, SIMONE DEHM<sup>1</sup>, RALPH KRUPKE<sup>1,2</sup>, MANUEL KONRAD<sup>1</sup>, WOLFGANG WENZEL<sup>1</sup>, ANKUSH KASHIWAR<sup>1,2</sup>, and HORST HAHN<sup>1,2</sup> — <sup>1</sup>Karlsruhe Institute of Technology, Germany  $^{\rm 2}{\rm Technical}$  University Darmstadt, Germany

In situ TEM techniques have developed tremendously during the last decade, providing direct quantitative structural and morphological information to understand reactions and processes in nanocrystalline materials in response to an external stimulus. We will illustrate some of the possibilities in situ TEM provides to develop a deeper materials understanding and discuss the challenges in interpreting in situ results using two types of experiments:

1) Mechanical deformation of nanocrystalline Pd and Au thin films, directly imaging grain rotation and grain boundary migration during straining and relaxation, analyzing correlations between grains and a strong Bauschinger effect observed in these materials, looking at both local processes and a statistical ensemble.

2) Thermally induced growth of nanocrystalline graphene, investigating the mobility and reactions of small graphene flakes, in particular the role of defects in different types of merging and Ostwald-type ripening of graphene flakes, supported by molecular modelling.

The influence of sample preparation and, in particular, electron beam effects on processes, reactions and kinetics will be critically discussed, to derive the main materials properties from in situ TEM experiments.

### MM 28.6 Wed 17:15 H45

TEM in-situ investigation of relaxation and dynamics in amorphous FeNiP nanorods — •Katharina Spangenberg, Sven HILKE, MARTIN PETERLECHNER, and GERHARD WILDE - University of Münster, Institute of Material Physics

The method of electron correlation microscopy (ECM) using transmission electron microscopy (TEM) has been presented by He et al. to investigate dynamics and relaxation phenomena at the atomic scale. Using conventional TEM tilted dark field, the method has the ability to spatially resolve the dynamical processes in the supercooled liquid region of an amorphous alloy [1]. In the present study, the influence of the electron dose rate and non-equilibrium dynamics of a morphous FeNiP in nanostructured confinement are investigated at room temperature. A minimum dose rate is estimated to ensure proper signal-tonoise ratio. Non-equilibrium dynamics were investigated and analysed using the time autocorrelation function g2 ( $\Delta t$ ) which can be fitted using a Kohlrausch-Williams-Watt (KWW) expression. ECM is used to calculate spatial distributions of relaxation times, represented by  $\tau$ -maps. The FeNiP glass exhibits a phase separation, e.g. upon heat treatment. EDX measurements reveal a homogeneous composition or a bamboo structure of amorphous Fe-rich and Ni-rich layers. By comparing the atomic fluctuations with the local phase composition, a relation between phase separation and the time scales of the heterogeneous dynamics can be discussed.

[1] P. Zhang, J. J. Maldonis, Z. Liu, J. Schroers, & P. M. Voyles, Nature communications, 9(1), 1129, 2018

 $\begin{array}{ccc} MM \ 28.7 & Wed \ 17:30 & H45 \\ \textbf{Fluctuation electron microscopy on PdNiP} & & \bullet \text{Farnaz Abdullahzadeh Davani}^1, \ Sven \ Hilke^1, \ Harald \ Rösner^1, \ David \end{array}$ 

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Metallic glasses are of interest due to their high strength and hardness. Plastic deformation at low and moderate temperatures is known to lead to localized shear with the formation of so-called shear bands. In this work, a Pd40Ni40P20 bulk metallic glass (BMG) was produced by ingot copper mold casting in a melt spinner under argon atmosphere. Plastic deformation of differently center edge notched beams was imposed under 3-point bending and in-situ monitored by optical microscopy. Undeformed and deformed states are compared using conventional and scanning transmission electron microscopy including high-angle annular dark-field imaging. Nano-beam diffraction patterns were collected and analyzed using variable resolution fluctuation electron microscopy. New insights with respect to structural changes induced by plastic deformation in bulk metallic glasses are discussed.