MM 32: Topical Session: TEM-Symposium - Structure-Property / In-Situ I

Time: Wednesday 10:15–11:30 Location: H25

Topical Talk MM 32.1 Wed 10:15 H25

Structure and deformation processes of nanocrystalline metals characterized by ACOM-STEM in combination with in-situ straining — Aaron Kobler[1,2], Horst Hahn[1,2], and •Christian Kübel[1,3] — [1]Karlsruhe Institute of Technology (KIT), Institute of Nanotechnology, 76021 Karlsruhe, Germany — [2]Technische Universität Darmstadt (TUD), KIT-TUD Joint Research Laboratory Nanomaterials, 64287 Darmstadt, Germany — [3]Karlsruhe Institute of Technology (KIT), Karlsruhe Nano Micro Facility, 76021 Karlsruhe, Germany

Understanding the deformation mechanisms in nanocrystalline (nc) metals and alloys is crucial for improving their performance and stability as needed for technical applications. In-situ deformation studies using XRD have contributed significantly to our current understanding of nc metals. However, it is difficult to understand the local processes and thin-film properties based on bulk measurements. These local processes are traditionally investigated using classical BF/DF-TEM. As an alternative, automated crystal orientation mapping (ACOM) inside a TEM has recently been developed for nanocrystalline materials as it allows identification of the crystallographic orientation of all crystallites and detects the CSL special boundaries within the imaged area. We have implemented ACOM on a FEI Tecnai F20 in micro-probe (up) STEM mode, that allows us to also acquire (fast) STEM reference images. We combined this ACOM-STEM imaging with in-situ straining inside the TEM using Hysitron*s Picoindenter. First investigations were conducted on magnetron sputtered Au samples and deformation of these films inside the TEM allowed us to follow the process of grain growth, grain rotation and (de)twinning in nc Au with increasing strain. New results will discuss the influence of alloying content in PdxAu1-x on the deformation behavior.

MM 32.2 Wed 10:45 H25

Grain size reduction by heating in nanocrystalline FeAl - a mystery solved by TEM — Christoph Gammer, Clemens Mangler, •Hans-Peter Karnthaler, and Christian Rentenberger — Universität Wien, Physik Nanostrukturierter Materialien, Boltzmanngasse 5, 1090 Wien, Austria

Bulk nanocrystalline materials show improved mechanical properties as compared to their coarse grained counterparts. Due to the high density of grain boundaries a thermodynamic driving force for grain growth exists and therefore, the thermal stability of a nanograined structure is usually low. In contrast, we show that in the present case of intermetallic FeAl made nanocrystalline by severe plastic deformation, a reduction of the grain size is occurring by heating connected with an increase in hardness [1]. High pressure torsion deformation of B2 ordered FeAl leads to a nanocrystalline material with strongly reduced long-range order. The samples show nanosized grains (about 70nm) with a high defect density and highly irregular grain boundaries. In-situ and post mortem TEM of thermally treated samples reveal a recurrence of the B2 superstructure [2], the re-arrangement of dislocations and what is rather unexpected, a reduction in grain size by a factor of 2. It is proposed that the decrease of the grain size during heating is linked to the recurrence of the long-range order by the interaction of the dislocations with the growing ordered domains. Therefore, subgrain boundaries turn into grain boundaries at a temperature too low for grain growth. [1] C. Mangler et al. Acta Mater 58 (2010) 5631. [2] C. Gammer et al. Scripta Mater 65 (2011) 57.

MM 32.3 Wed 11:00 H25

Combined use of ex-situ and in-situ TEM for the analysis of the nanocrystallization of bulk amorphous NiTi — •Martin Peterlechner[1], Thomas Waitz[2], and Gerhard Wilde[1] — [1]Institute of Materials Physics, WWU Münster, Germany — [2]Physics of Nanostructured Materials, University of Vienna, Austria

A NiTi shape memory alloy was subjected to severe plastic deformation by repeated cold rolling (RCR). RCR yields almost complete amorphization. A small volume fraction of nanocrystalline debris is embedded heterogeneously in the amorphous matrix. Upon annealing nanocrystallization occurs. The crystallization kinetics were analyzed using ex-situ and in-situ transmission electron microscopy (TEM) experiments. Ex-situ TEM investigations were carried out of bulk samples crystallized at 307°C (270 min) in a calorimeter that allows precise control of the temperature. Ex-situ TEM specimens were analyzed at room temperature. Additional, in-situ TEM heating was carried out at a nominal temperature of 307°C. From in-situ TEM it can be concluded that hard-impingement is present. Therefore, the grain sizes formed by ex-situ nanocrystallization can be used to analyze the kinetics based on hard impingement models. The crystallization causes grains with a bimodal size distribution. This is explained considering the heterogeneous distribution of nanocrystals left in the amorphous matrix that can act as nucleation sites of new grains. Since the number density of the nanocrystalline debris depends on the degree of deformation, both the degree of the deformation and the crystallization temperature can be used to tailor the grain size.

MM 32.4 Wed 11:15 H25

TEM study of Co_3Ti made amorphous by high pressure torsion — •David Geist, Christoph Gammer, Hans-Peter Karnthaler, and Christian Rentenberger — University of Vienna, Physics of Nanostructured Materials, 1090 Vienna, Austria

Amorphous structures can be achieved by a solid state transformation of the crystalline structure using severe plastic deformation. This has been successfully achieved by applying high pressure torsion (HPT) deformation in the case of $L1_2$ ordered intermetallic Zr_3Al [1,2]. In the present work the evolution, structure and stability of the amorphous state of bulk Co_3Ti were studied on multiple length scales using a combination of scanning (SEM) and high resolution transmission electron microscopy (HRTEM) methods. $L1_2$ ordered Co-23at.%Ti samples were deformed by HPT to different degrees of deformation. The SEM investigations using back scattered electrons reveal that the material shows a tendency to amorphization localized in the form of bands. The TEM results of the amorphous regions show that a high density of nanosized crystals having a crystalline structure different from the initial material is embedded in the amorphous phase. Electron diffraction and HRTEM reveal the presence of nanocrystals with Co_2Ti structure showing a modified stacking sequence. Their structure is compared to that resulting from devitrification of the amorphous regions during thermal treatment. [1] D. Geist, C. Rentenberger, H. P. Karnthaler. Acta Mater. **59** (2011) 4578. [2] D. Geist, S. Ii, K. Tsuchiya, H.P. Karnthaler, G. Stefanov, C. Rentenberger. JAllComp **509** (2011) 1815.