MM 23: Electron Microscopy I - Nanomaterials

Time: Tuesday 10:15–11:30 Location: IFW D

MM 23.1 Tue 10:15 IFW D

Analytical EDX studies at different temperatures of Boron/Ni composite nanowires — •Daniela Sudfeld, Bastian Barton, Oleg Lourie, and Bert Freitag — FEI Electron Optics B. V., Eindhoven, The Netherlands

Analytical EDX studies at different temperatures were performed in a S/TEM, FEI Talos[TM] with ChemiSTEM[TM] Technology [1]. Fast chemical maps of B/Ni composite nanowires on nanometer scale by EDX (energy-dispersive X-ray spectroscopy) were done with the new Velox[TM] software. Successful synthesis of crystalline nanowires composed of the refractory light materials such as Boron can enable novel applications for nanoelectronics [2-4]. Boron/Nickel composite nanostructures were prepared by a CVD-based synthetic procedure with a Ni-based compound catalyst; naturally blended with high conductivity and refraction index. The properties of this binary nanomaterial at room temperature are compared to those achieved from heating experiments with temperatures up to 1000 deg C. 2D-3D EDX chemical mappings show clearly the core-shell structure of the wires: B in the shell and Ni in the core. This is amplified at elevated temperatures of ca. 500 deg C. At ca. 1,000 deg C EDX maps reveal also that Ni vanishes from the core, leaving behind hollow B nanowire (nanotube) structures. [1] P. Schlossmacher et al., Microscopy Today 18(4) (2010) 14. [2] CJ Otten, et al., J Am Chem Soc. 2002 May 1;124(17):4564. [3] D. Wang et al., APL 2003, 183(25):5280. [4] W. Ding et al., Mech, Comp. Sci. and Techn. 2006, 66:1109.

MM 23.2 Tue 10:30 IFW D

Evolution of the microstructure of amorphous FeNiP nanowire arrays upon in-situ annealing in TEM — •Nina Winkler, Martin Peterlechner, and Gerhard Wilde — Institute of Materials Physics, Westfälische Wilhelms-Universität Münster, Wilhelm-Klemm Str. 10, 48149 Münster, Germany

In this work, an amorphous soft magnetic material is studied in confined dimensions regarding its crystallization behavior. Therefore, porous Anodic Alumina Oxide (AAO) is used because it enables the control of regularity and shape of nanostructures via its pores which are uniform in length and uniform in diameter. The fabrication of amorphous nanowire arrays by electrodeposition using AAO will be addressed. Upon in-situ annealing in the transmission electron microscope (TEM) the microstructural evolution could be monitored. Long range diffusion was observed following a phase separation of FeNiP in multilayers of FeNi and FeNiP phases. The impact of heating rates on the phase evolution is studied by controlled annealing with different heating rates of the FeNiP nanowires in atmosphere using a Differential Scanning Calorimeter (DSC). The magnetic properties of the nanowire arrays have been characterized with a Vibrating Sample Magnetometer (VSM). The results will be discussed with respect to the different microstructures of the nanowire arrays caused by the different thermal treatments.

MM 23.3 Tue 10:45 IFW D

Nucleation of embedded Pb nanoparticles in an Al(Ga) matrix — •Anna Moros[1], Sorin Lazar[2], Harald Rösner[1], Peter Schlossmacher[2], and Gerhard Wilde[1] — [1]Institute of Materials Physics, Wilhelm-Klemm-Straße 10, 48149 Münster, Germany — [2]FEI Company, Achtseweg Noord 5, 5600 KA Eindhoven, The Netherlands

Undercooling of the liquid phase is often observed for most liquid pure materials. This indicates the existence of a large energy barrier to nucleate the solid phase. Heterogeneous nucleation at interfaces is generally favoured, since the nucleation barrier is reduced due to pre-existing catalytic substrates. Confined systems such as nanometer-sized particles embedded in a matrix have large specific interface areas that might possibly act as heterogeneous nucleation sites during solidification upon cooling, specifically since both, Pb and AlGa crystallize in fcc structure. In the present work, nanoscaled Pb inclusions embedded in AlGa matrices were processed by melt spinning technique. Various Ga quantities were alloyed into the matrix, since Ga is miscible with Al and immiscible with Pb. By means of differential scanning calorimetry, large undercoolings of Pb nanoparticles in (Al99-xGax)Pb1 alloys were observed that increased with increasing amounts of Ga. At the same time, nanoscale chemical analyses using the SuperX detector implemented in a FEI Titan 60-300 microscope were performed and Ga segregation at the interfaces of the embedded Pb nanoparticles with AlGa-matrix was observed. In this contribution, the correlation between the segregation of Ga at the particle-matrix interface and the resulting undercooling of the nanoscaled Pb particles will be discussed.

MM 23.4 Tue 11:00 IFW D

Characteristics of shear bands in metallic glasses investigated by analytical TEM — •Vitalij Schmidt, Harald Rösner, Martin Peterlechner, and Gerhard Wilde — Institut für Materialphysik, Westfälische Wilhelms-Universität Münster, Wilhelm-Klemm-Str. 10, 48149 Münster

In metallic glasses the entire deformation flow is restricted to narrow regions called shear bands. They are associated with a structural change compared to the surrounding matrix due to local distortion, most likely resulting in enhanced free volume. Therefore deformation properties of metallic glasses are determined by shear band characteristics which are a current object of research.

We present investigations of shear bands with a new approach that combines high angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) with electron energy loss spectroscopy (EELS) and energy dispersive x-ray (EDX) analysis. It is thereby possible to correlate dark-field intensity and local density by $\Delta\rho = \frac{I_{\mathrm{SB}}-I_{\mathrm{M}}}{I_{\mathrm{M}}}$, where $\Delta\rho$ is the difference in density, I_{SB} and I_{M} are the HAADF intensities of the shear band and the matrix, respectively.

Cold-rolled $Al_{88}Y_7Fe_5$ melt-spun ribbons show shear bands having frequent contrast changes. Density measurements approve a continuous variation throughout the band*s length implying different propagation velocities within the bright and dark parts.

MM 23.5 Tue 11:15 IFW D

Density of Grain Boundaries — •Yulia Buranova, Sergiy Divinskiy, Harald Rösner, and Gerhard Wilde — Institut für Materialphysik, Westfälische Wilhelms-Universität Münster, Wilhelm-Klemm-Str. 10, D-48149 Münster, Germany

In this work we present the results of atomic simulations of different Grain Boundaries (GB) and the corresponding excess free volume defined as a relative density. For this purpose, an image analysis tool was designed that allows analyzing the local mass density on the basis of simulated (or experimental) high-resolution transmission electron microscopy (HRTEM) images. Thereto the intensities of the GB areas were compared with that of the grain interiors and the difference was identified as the density change.

In total 24 different Symmetrical Tilt GB (STGB) were simulated. The density change in the STGB was estimated to be 5-6%. Experimentally, aluminum was investigated using HRTEM. Calculations showed that this method works for the pure samples with thicknesses up to 15nm including aluminum oxide layers. The correctness of the method is evaluated for different artificial configurations including chains of vacancies and solute atoms.The results and the method are discussed.