# MM 32: Microstructure and Phase Transformations - detection methods

Time: Tuesday 11:45–13:15 Location: IFW B

MM 32.1 Tue 11:45 IFW B

**In situ investigation of the Microwave-Assisted Solvothermal Synthesis process by Small-Angle X-ray Scattering** — •Eike Gericke[1], Robert Wendt[1], Armin Hoell[2], Dragomir Tatchev[3], Simone Raoux[2], and Klaus Rademann[1] — [1]Humboldt-Universität zu Berlin, Berlin, Germany — [2]Helmholtz-Zentrum Berlin, Berlin, Germany — [3]Bulgarian Academy of Sciences, Sofia, Bulgaria

Microwave-assisted solvothermal syntheses (MWASS) improve well-established solvothermal syntheses for the preparation of monodisperse and crystalline metal and metal oxide nanoparticles. MWASS allows a precise control of pressure and temperature inside a sealed reaction vessel. The advantage in contrast to conventional heating is the efficient internal heating by direct coupling of MW energy into the precursor solution, allowing high heating rates with small thermal gradients. Hence, the reaction times are decreased significantly and can be described by the Arrhenius law. Understanding the reaction mechanisms inside a MWASS reactor is important to influence and optimize the reaction process accordingly. The reaction kinetics are in a scale of seconds. So, the nucleation and growth processes taking place in a sealed high-pressure reactor have to be monitored under extreme conditions. A sample environment, elegant adjusted to these conditions will be presented, usable for in situ SAXS. Extension by WAXS and XAS or UV-vis and Raman are possible. It resists harsh chemical conditions and pressures above 30 bar. Results from experiments at Elettra and ESRF will be presented allowing unique insights in the particles growth mechanisms during a MWASS.

MM 32.2 Tue 12:00 IFW B

**Time resolved diffraction on cooling of Ti-Fe alloys solidified from different undercooling levels** — •Olga Shuleshova[1], Ivan Kaban[1], Dirk Holland-Moritz[2], Jan Gegner[2], Junhee Han[1], Norbert Mattern[1], and Jürgen Eckert[3] — [1]Institute for Complex Materials, IFW Dresden, Germany — [2]Institute of Materials Physics in Space, DLR, Cologne, Germany — [3]Montan-University Leoben, Austria

Melt undercooling is a necessary precondition to begin solidification. With help of the containerises processing, realised in different levitation techniques, undercoolings of the order of several hundreds degrees are easily achieved in many pure metals as well as in metallic alloy systems. With increasing undercooling a profound effect of solute trapping have been observed in alloys. Time resolved diffraction of synchrotron X-rays on levitated metallic droplets allows to quantitatively analyse the level of supersaturation present in the sample immediately after solidification and trace its further evolution as sample is cooled down. Applied to the Ti-Fe system this method have shown a non-intuitive results due to coupling with other effect of undercooling - the microstructure refinement. Financial support by DFG under project No. SH 578/1-1 is gratefully acknowledged.

MM 32.3 Tue 12:15 IFW B

**Positron-$\mu$m-beam for the Coincident Doppler Broadening Spectrometer at NEPOMUC** — •Thomas Gigl, Marcel Dickmann, Markus Reiner, Benjamin Rienäcker, Matthias Thalmayr, and Christoph Hugenschmidt — Heinz Maier-Leibnitz Zentrum (MLZ) and Physik Department E21, Technische Universität München, Lichtenbergstr. 1, 85748 Garching, Germany

The positron beam facility NEPOMUC (Neutron induced Positron Source Munich) at the neutron source FRM II provides a monoenergetic positron beam with high intensity of $10^9$ moderated positrons per second. A new CDB spectrometer was set into operation which enables defect studies by conventional Doppler broadening spectroscopy (DBS) and element-specific measurements with coincident DBS (CDBS) at the near surface and in the bulk of a sample with a positron implantation energy of up to 30 keV. According to first spatially resolved measurements the CDB Spectrometer provides a lateral resolution of 200 $\mu$m. With an additional 100 nm thin Ni(100) remoderation foil in transmission geometry a resolution of 50 $\mu$m could be measured. In order to achieve a high yield of re-emitted moderated positrons, the Ni foil has to be annealed and surface contaminations such as C and O have to be removed. For this purpose the foil was heated to about 400°C in a H-atmosphere of about $10^{-3}$ mbar. After this procedure and cooling down to room temperature, the moderation efficiency of the Ni(100) foil could be measured to about 10%. Financial support by BMBF (project no. 05K10WOB) is gratefully acknowledged.

MM 32.4 Tue 12:30 IFW B

**The lattice strain in single-phase high-entropy alloys** — •Junhee Han[1], Pramote Thirathipviwat[1,2], Jens Freudenberger[1], Jozef Bednarcik[3], Norbert Mattern[1], and Thomas Gemming[1] — [1]IFW Dresden, PO Box 270116, D-01171 Dresden, Germany — [2]TU Dresden, Institute of Materials Science, 01062 Dresden, Germany — [3]DESY Photon Science, Notkestraße 85, 22607 Hamburg, Germany

The formation of single-phase solid solutions in high-entropy alloys (HEAs) consisting of multiple principal elements has led to the proposition that the lattices of HEAs must be severely distorted. Severe lattice distortion constitutes one of the key features characterizing high-entropy alloys and their enhanced mechanical properties. However, quantitative determination of the lattice distortions have only rarely been investigated. In order to address this issue, we employed means of X-ray diffraction (XRD) studies to investigate both long-range and atomic-scale distortions in the fcc-structured HEA and its binary, ternary and quaternary subsystems. The long-range and the local lattice strain were determined from the peak broadening analysis of X-ray diffraction and pair distribution functions (PDF). Particular attention is paid to the relative difference in the lattice distortions between the HEA and their subsystems including unary fcc-Ni. The HEA exhibited only slight changes in the long-range and local lattice strain compared to those of the subsystems. The results in this study indicate that the lattice strains in the equiatomic HEA is only slightly distorted away from the average fcc structure.

MM 32.5 Tue 12:45 IFW B

**Imaging and kinetics of MgH2 formation** — •Efi Hadjixenophontos[1], Manuel Roussel[1], Toyoto Sato[2], Patrick Stender[1], Shin-ichi Orimo[2,3], and Guido Schmitz[1] — [1]Institut für Materialwissenschaft (IMW) University of Stuttgart, Heisenbergstrasse 3, 70569 Stuttgart GERMANY — [2]Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 JAPAN — [3]WPI-Advanced Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 JAPAN

Storage in metal hydrides is presented as one of the solutions to solve the hydrogen storage problem. MgH2 is one of the ideal materials, studied intensively for the hydrogen fuel based economy. This reversible system has however some drawbacks. Hydrogen and Mg are strongly bonded by a H bond, and this makes the stable compound unpractical for commercial use. During this work Mg thin films will enable us to monitor the growth process of the hydride and study the mechanism of hydride formation. Pd is used as a catalyst coating for decreasing the pressure and temperature of hydrogenation to 5bars and 100°C. The hydride formation has been followed by in-situ XRD characterization. Microscopic imaging of the co-existence of MgH2 and Mg is presented by SEM and TEM. The microstructure change is clearly visible in the micrographs, despite the fact that electron microscopy damages the hydride phase. These combined techniques are a great way to follow the kinetics of hydride formation within the layer,and study further the diffusion coefficients and mechanism of hydrogenation at 200°C and 300°C and at different pressures (1-100bars).

MM 32.6 Tue 13:00 IFW B

**Trajectory-based reconstruction in atom probe tomography** — •Daniel Beinke, Christian Oberdorfer, and Guido Schmitz — Institut für Materialwissenschaft, Universität Stuttgart

Atom probe tomography provides detailed three dimensional chemical information of a wide range of materials by the field evaporation of needle-shaped specimens. The field-evaporated ions are accelerated towards a detector and the time-of-flight as well as the hit position is measured. The reconstruction of the recorded detector events is critical in order to access the three dimensional information of the analyzed material. The standard reconstruction approach, introduced by Bas et al. [1], is based on a linear point projection between the measured detector position and a projection point located on the tip axis. The most remarkable success of this technique is the ability to recover lat-

tice planes. However, characteristic artifacts occur, especially in the case of significantly different evaporation fields, i.e. near grain boundaries or in multicomponent layer structures. In this work, a concept for a reconstruction technique based on the calculation of realistic ion trajectories is presented. In a first step, the approach is tested on a rigid lattice [2]. Afterwards, the technique is expanded in order to deal with a limited detector efficiency and an unrestricted set of possible positions for the reconstruction of single atoms.

[1] P. Bas et al., Appl Surf Sci 87 (1995) 298-304. [2] D. Beinke et al., Ultramicroscopy 165 (2016) 34-41.