MM 23: Phase Transformations I

Time: Wednesday 11:00–13:15 Location: IFW D

MM 23.1 Wed 11:00 IFW D

Junction controlled grain growth: evolution equations and grain size distributions — •Peter Streitenberger and Dana Zöllner — Institut für Experimentelle Physik, Abteilung Materialphysik, Otto-von-Guericke-Universität Magdeburg, Universitätsplatz 2, D-39106 Magdeburg

Normal grain growth is essentially controlled by the grain boundaries. However, the other structural elements of a 3D grain network (triple lines and quadruple points) may have likewise a strong effect on the growth kinetics but at very small grain sizes. For a systematic estimation of the influence of the energetic and kinematic properties of boundary junctions on grain growth each type of junction is assigned an own specific energy and mobility. By application of a thermodynamic variation principle a general grain evolution equation is derived.

Apart from the already known size parameters associated with finite boundary junction mobilities two further size parameters associated with non-vanishing specific energies of triple lines and quadruple points enter the calculation. Consequently, apart from linear and exponential kinetics already considered by Gottstein, Shvindlerman and others, six further types of growth kinetics can be identified at very small grain sizes. Each type of growth kinetics is characterised by a separable scaling form of the grain evolution equation from which analytical expressions of the corresponding self-similar size distribution function are derived. The obtained size distributions of triple and quadruple junction limited grain growth are clearly shifted to smaller relative sizes indicating a strong population enhancement in this range.

MM 23.2 Wed 11:15 IFW D

In-situ Messungen von magnetisch getriebener Korngrenzenbewegung in Zink-Bikristallen — •Christoph Günster — RWTH Aachen, Aachen, Deutschland

In der vorliegenden Arbeit wurde die magnetisch getriebene Korngrenzenbewegung in hochreinen (99.995%) Zink-Bikristallen untersucht. Die Beweglichkeit von <10-10>-Kippkorngrenzen mit Desorientierungswinkeln zwischen 60° und 90° wurden bestimmt. Aufgrund der magnetischen Anisotropie von Zink, können unter dem Einfluß eines externen Magnetfeldes Korngrenzen (KGen) von einer magnetischen treibenden Kraft angetrieben werden. Die Ergebnisse bestätigen, daß eine ausgeprägte Abhängigkeit der KGen-Beweglichkeit zur Desorientierung besteht. Darüber hinaus besaßen die gemessenen Bewegungsaktivierungsparameter für ebene, asymmetrische KGen deutlich höhere Werte, als dies in der Vergangenheit für krümmungsgetriebenen KGen in Zink-Bikristallen berichtet wurde. Nach weiteren Experimenten an symmetrischen KGen wurden deutlich niedrigere Aktivierungsparameter festgestellt als bei unseren Experimenten an asymmetrischen KGen, was einen Hinweis auf die Existenz einer ausgeprägten Inklinationsabhängigkeit der Beweglichkeit von Kipp-KGen in Zink darstellt. Weiterhin unterstützen diese Daten unsere Ergebnisse aus früheren Experimenten an symmetrischen und asymmetrischen Kipp-KGen mit Bi-Bikristallen. Weiterhin sind diese Ergebnisse ein Hinweis auf das Ablaufen jeweils unterschiedlicher atomistischer Mechanismen bei der Bewegung von symmetrischen und asymmetrischen Kipp-KGen in Zink.

MM 23.3 Wed 11:30 IFW D

In situ three-dimensional investigation of Ostwald ripening — •Thomas Werz[1], Uwe Wolfram[2], and Carl E. Krill III[1] — [1]Institute of Micro and Nanomaterials, University of Ulm, Germany — [2]Institute of Orthopaedic Research and Biomechanics, University Hospital Ulm, Germany

Owing to their destructive nature, conventional techniques for investigating Ostwald ripening — the thermally induced coarsening of two-phase microstructures — do not allow repeated observation of the same volumetric region in a sample. In this work, we employed laboratory microcomputed tomography (microCT) to carry out nondestructive, three-dimensional characterization of an Al-5 wt.% Cu alloy undergoing Ostwald ripening. The sample was repeatedly annealed at 630°C, inducing a semisolid state with a volume fraction $V_V = 60\%$ of the coarsening (solid) phase. Each annealing step was followed by a microCT scan performed at room temperature. When semisolid, the system consists of Al-rich grains surrounded by a liquid phase of higher Cu content. Tomographic contrast between the two phases arises from their differing attenuation factors for x-ray radiation. During cooling, the liquid phase tends to retreat into the triple junctions, resulting in incomplete coverage of the boundaries separating the coarsening grains. Correction for the latter effect was accomplished by a watershed-transform-based image processing routine, enabling reliable grain segmentation and the evaluation of local and global microstructural parameters in 3D. These can, in turn, be compared to theoretical and simulation results.

MM 23.4 Wed 11:45 IFW D

Microstructural investigation and thermal stability of ball-milled Fe-Cu — •Catharina G. Wille and Tala'at Al-Kassab — King Abdullah University of Science and Technology (KAUST), Materials Science and Engineering, Thuwal 23955-6900, Kingdom of Saudi Arabia

The microstructural changes upon heat treatment in ball-milled Fe-10at.%Cu powders after different milling times from 2 to 10h were observed by means of transmission electron microscopy (TEM). This heat treatment was performed isochronally inside a differential scanning calorimeter (DSC).

The heat release calculated from the DSC curves will be correlated to the observed microstructural changes. Additionally the positions of the exothermic peaks and the respective stored enthalpies will be compared to the ones obtained within earlier works and especially their conclusions on phase separation - accompanied by grain growth and strain release.

For the minority component copper as well as for the impurity oxygen the segregation and precipitation behaviour in connection with grain growth were investigated by atom probe tomography (APT).

MM 23.5 Wed 12:00 IFW D

Advanced Characterization of High Performance Permanent Magnets for Hybrid Electric Vehicles — •Thomas George Woodcock and Oliver Gutfleisch — IFW Dresden, Institute for Metallic Materials, PO Box 270116, 01171 Dresden, Germany

NdFeB sintered magnets have recently found important new applications in electric motors for hybrid electric vehicles (HEV) and in the generators used in wind turbines. The operating temperature of the electric motor in HEV is typically approx. 180°C and therefore magnet grades with high Dy content are required in order to provide a sufficiently large coercivity at such temperatures. The limited availability of Dy has led to a very high and somewhat fluctuating price of that element. Significant research effort has therefore recently been put into reducing the Dy content or eliminating the need for Dy in NdFeB sintered magnets for high temperature applications. In addition to various experimental routes toward achieving this, novel approaches yielding detailed microstructural characterisation are required in order to bring greater understanding of coercivity mechanisms. High resolution SEM and TEM, magneto-optical Kerr microscopy and MFM are routinely used to examine a range of NdFeB materials. Recent developments include the use of aberration-corrected TEM to obtain highest spatial resolution images and the application of electron backscatter diffraction (EBSD) to obtain crystallographic orientation data on a local scale from all the phases present in the microstructure. The combination of EBSD and EDX with high resolution serial sectioning yields 3D orientation and chemical data which will also be discussed.

MM 23.6 Wed 12:15 IFW D

Surface studies of Fe polycrystals with LEEM — •Benjamin Borkenhagen[1], Thorsten Franz[2], Gerhard Lilienkamp[1], and Winfried Daum[1] — [1]IEPT, TU Clausthal, Leibnizstr. 4, 38678 Clausthal-Zellerfeld — [2]ELMITEC GmbH, Albrecht-von-Grodeck-Str. 3, 38678 Clausthal-Zellerfeld

Low energy electron microscopy (LEEM) has been proven a versalite tool for studies on single crystal surfaces. In this contribution, we present the application of LEEM to study the microstructure and processes on surfaces of polycrystals. The additional capability of this microscope to acquire low energy electron diffraction patterns of crystallites (μ-LEED) allowed us to distinguish between different crystallographic surface structures. We observed atomically flat terraces separated by monoatomic steps as well as facetted surfaces. Islands with regular shapes were detected at the surfaces of some crystallites. In

situ investigations during heating of the polycrystal showed that these islands were bulk impurities which segregated to the surface, or diffused back to the bulk depending on temperature. Parallel imaging of the whole field by LEEM enabled the observation of dynamic processes such as grain boundary motion and segregation processes at elevated temperatures.

MM 23.7 Wed 12:30 IFW D

Ground state structure in Au-50 at.% Pd — •Cédric Sax[1], Bernd Schönfeld[1], and Andrei Ruban[2] — [1]LMPT, Department of Materials, ETH Zurich — [2]KTH Stockholm, Sweden

From experiment no ordered structure is known for the bulk Au-Pd system. Electronic structure calculations indicate the CH structure for 1:1 stoichiometry, diffuse scattering also allows plausible candidates for ordered structures to be discussed. In this work diffuse x-ray scattering was measured at room temperature from a single crystal of Au-48 at.% Pd. The crystal was aged at 703 K to set up a state of thermal equilibrium. The short-range order scattering is characterized by $2k_F$ maxima where k_F is the Fermi wave vector along <110>. From the separated short-range order scattering effective pair interaction parameters were determined. Ordering energies were calculated for several $L1_0$-based long-period superstructures (LPS) as LPS are favorably formed in systems with $2k_F$ maxima. These results from diffuse scattering will be compared with those from recent electronic structure calculations.

MM 23.8 Wed 12:45 IFW D

Undercooling and solidification of Ni_2B under different convective flow conditions — •Sven Binder[1,2], Jianrong Gao[3], and Dieter M. Herlach[1] — [1]Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt, 51170 Köln, Germany — [2]Institut für Festkörperphysik, Ruhr-Universität Bochum, 44780 Bochum, Germany — [3]Key Laboratory of Electromagnetic Processing of Materials, Northeastern University, Shenyang 110004, China

We investigate the kinetics of crystal growth by measurements of the dendrite growth velocity as a function of undercooling during non-equilibrium solidification. Measurements are conducted under different conditions of convection. The liquid samples are levitated and undercooled in strong alternating electromagnetic fields leading to forced convection. Inductive stirring is avoided by processing the samples in a glassy slag where only natural convection is present. Forced convection and natural convection can be reduced by performing undercooling experiments in reduced gravity. The experimental results obtained under different conditions are compared to each other in order to investigate the influence of convection on the growth dynamics of dendrites in undercooled melts. The congruently melting compound Ni_2B is chosen as a suitable sample system. It forms an intermetallic phase with growth velocities that are comparable to the fluid flow velocities in electromagnetically levitated melts. The results are analyzed within dendrite growth models and reveal that the growth velocity is essentially influenced by forced convection in strong electromagnetic fields. The present work is supported by DFG under contract HE1601/25.

MM 23.9 Wed 13:00 IFW D

Phase Formation and Martensitic Transformation of Cast Cu-Zr-Co Alloys — •Fatemeh A. Javid[1], Norbert Mattern[1], and Jürgen Eckert[1,2] — [1]IFW Dresden, Institute for Complex Materials, Dresden, Germany — [2]Technical Uneversity, Dresden, Germany

The phase formation and martensitic transformation of $Cu_{50-x}Zr_{50}Co_x$ ($x = 0, 2, 5, 7.5, 10, 20$ at.%) melt-spun and suction-cast bulk specimens has been studied. X-ray analysis shows that cobalt affects the eutectoid reaction $CuZr \leftrightarrow Cu_{10}Zr_7 + CuZr_2$ of the binary Cu-Zr system and shifts it to lower temperatures. The results indicate that in compositions with at least 5 at. % cobalt, the glass crystallizes directly into B2(Cu,Co)Zr and this phase is the equilibrium phase at room temperature. An extended solid solution $Cu_{50-x}Zr_{50}Co_x$, cobalt between 5 and 50 at.% is indicated by the lattice parameter of the B2-type phase versus Co-content. The X-ray analysis of bulk specimens shows that in compositions with more that 10 at. % of cobalt, the B2(Cu,Co)Zr phase is coming from the liquid directly and this is the main phase at room temperature while compositions with less that 10 at.% of cobalt contain monoclinic CuZr phases as the main phase. Martensitic transformation of bulk samples was investigated with differential scanning calorimeter and the results show that cobalt shifts the transformation temperatures to lower temperatures. The martensite transformation start temperature lowers from about 452 K for $x = 0$ to room temperature for $x = 7.5$.