MM 29: Microstructure and Phase Transformations

Sessions: Microstructure and Phase Transformations I and II

Time: Wednesday 15:00–18:15 Location: H46

MM 29.1 Wed 15:00 H46

Abnormal grain growth in friction stir welded aluminium with zirconium additives — •Moritz Winter[1], Maximilian Gnedel[1], Amanda Zens[2], Ferdinand Haider[1], and Michael F. Zäh[2] — [1]Chair for Experimental Physics I, University of Augsburg, Universitätsstraße 2, 86159 Augsburg (Germany) — [2]Institute for Machine Tools and Industrial Management, Technical University of Munich, Boltzmannstraße 15, 85748 Garching (Germany)

Friction Stir Welding (FSW) is a well-established solid-state joining process for materials such as aluminium. In Al-Li-Cu alloys, such as AA2195 aluminium, abnormal grain growth (AGG) can be a crucial issue during post-processing of the produced welds, whilst the alloy contains zirconium for grain boundary stabilisation. Both, the microstructural stability during subsequent heat treatment as well as the mechanical properties in general can be optimized by intermixing specific micrometer-sized metal powders as well as metal foils. By dispersing additional zirconium powder inside a matrix of AA2195 aluminium during FSW, the formation of abnormal grains can be reduced, if suitable processing parameters are used. Using optical microscopy, the reduction of the secondary recrystallized area can be revealed. In addition, the formation of intermetallic phases, acting as pinning particles to hinder AGG, can be evaluated using transmission electron microscopy (TEM). Furthermore, electron backscatter diffraction (EBSD) provides insights on the changes of the crystallographic texture of the welding regions both post-weld and after subsequent heat treatment.

MM 29.2 Wed 15:15 H46

Reverse engineering the kinetics of grain growth from time-resolved 3DXRD measurements — •Mingyan Wang[1], Jules M. Dake[1], Søren Schmidt[2], and Carl E. Krill III[1] — [1]Ulm University, Germany — [2]Technical University of Denmark, Denmark

Though grain growth has been studied in polycrystalline materials for many years, our understanding of the underlying mechanisms remains incomplete. Even in the case of normal grain growth, computer simulations differ significantly from experimental findings. Instead of modeling this phenomenon using boundary parameters derived from physical principles, we decided to try out the opposite approach: that is, to work backwards from experimental measurements to the kinetics of boundary migration. Employing 3D x-ray diffraction (3DXRD) microscopy, we investigated grain growth in two different Al alloys. In a series of microstructural snapshots taken between isothermal annealing steps, we followed the morphology, misorientation and migration of thousands of grain boundaries (GBs) in a single sample. The results enable us to extract the dependency of reduced mobility (the product of GB mobility and energy) on GB misorientation. In one particular specimen, this dependency is consistent with expectations for normal grain growth, but, in the other case, we find evidence for abnormal kinetics that do not fit standard models.

MM 29.3 Wed 15:30 H46

Kinetic analysis of the precipitation sequence in dilute Al-Mg-Si alloys based on isothermal in-situ high-precision dilatometry measurements — •Robert Enzinger, Elisabeth Hengge, Wolfgang Sprengel, and Roland Würschum — Institute of Materials Physics, Graz University of Technology, Graz, Austria

The precipitation sequence in a dilute Al-Mg-Si alloy is studied utilizing recent progress in high-precision isothermal dilatometry, which yield access to tiny relative length changes $\Delta L/L$ in the range of 10^{-5} that occur on long time scales exceeding 10^5 s during isothermal ageing. For each forming phase, $\Delta L/L$-contributions arise from the volume excess due to the precipitates and the contraction of the matrix upon precipitation of solute atoms. In this way, the metastable coherent β''- and the semi-coherent β'-phase as well as the stable incoherent β-phase could unambiguously be detected on an absolute scale [1]. Reaction-rate analyses of the $\Delta L/L$ measurements based on the Johnson-Mehl-Avrami-Kolmogorov model yield the kinetic parameters for the formation of the β''-phase and either its transformation to or the formation of the β'-phase. By compiling all these findings, the industrial highly relevant time-temperature-precipitation diagrams become easily accessible, which for these types of alloys were so far hard to ascertain with common experimental techniques.

[1] E. Hengge, R. Enzinger, M. Luckabauer, W. Sprengel, and R. Würschum, Phil. Mag. Lett. (2018), DOI: 10.1080/09500839.2018.1542170

MM 29.4 Wed 15:45 H46

Mechanical dissolution of copper additions in aluminium by Friction Stir Processing — •Maximilian Gnedel[1], Amanda Zens[2], Ferdinand Haider[1], and Michael Friedrich Zaeh[2] — [1]Chair for Experimental Physics I, University of Augsburg, Universitätsstraße 2, 86159 Augsburg, Germany — [2]Institute for Machine Tools and Industrial Management, Technical University of Munich, Boltzmannstraße 15, 85748 Garching, Germany

Friction Stir Processing (FSP) is an established method for modifying properties in materials such as aluminium. Furthermore, the composition of the alloy can be changed by this technique. Intermixing specific micrometer-sized metal powders as well as metal foils helps to optimize both the microstructural stability during subsequent heat treatment, as well as the mechanical properties in general. Dispersing copper powder inside a matrix of AA1050 aluminium by FSP can produce a homogenous solid solution of the two elements, if suitable processing parameters are used. A substantial hardening effect is shown, due to both factors, refinement of the grain structure and solid solution hardening. By calculating the diffusion length and using the results of investigations based on energy-dispersive X-ray spectroscopy (EDS), it can be shown that the dissolution is mainly mechanically driven and not due to diffusion. Furthermore, transmission electron microscopy (TEM) provides insights on the formation of intermetallic phases during FSP, as well as after subsequent, targeted heat treatment. The results can be used in future studies to evaluate the properties of such highly deformed non-equilibrium alloys.

MM 29.5 Wed 16:00 H46

Study on nucleation rates and nucleation parameters of binary alloys — •Manoel da Silva Pinto, Martin Peterlechner, and Gerhard Wilde — Institute of Material Physics, Münster, Germany

Nucleation has an importance in diverse phenomena studied in a broad range of scientific areas. In material science, topics of special interest are, among others, new phase nucleation in alloys, solid-liquid interface energies, glass formability and possible formation of new phases in deep undercooled melts. Yet, only a few data is available on the general behavior of nucleation in alloy melts. With the help of a statistical approach related to survival analyses applied to calorimetric data of deeply undercooled alloys, we measure and analyze the nucleation rates of Au-Ge and Cu-Ge alloys for different compositions. Further parameters can be derived from this data and a comparison with the theoretical predictions from the classical nucleation theory of pure metals can be made. A dimensionless solid-liquid interface energy, the so called Spaepen factor, is determined in order to gather information about the possible nucleating phases. As a quantity depending on the crystallographic structure of the lattice formed during nucleation, the Spaepen factor could be used to describe the most probable nucleating phase and therefore the nucleation behavior in alloy melts.

MM 29.6 Wed 16:15 H46

Connectivity evolution during coarsening of nanoporous gold via kinetic Monte Carlo simulations — •Yong Li[1,2], Bao-Nam Dinh Ngô[2], Jürgen Markmann[1,2], and Jörg Weissmüller[1,2] — [1]Institute of Materials Physics and Technology, Hamburg University of Technology, Hamburg, Germany — [2]Institute of Materials Research, Materials Mechanics, Helmholtz-Zentrum Geesthacht, Geesthacht, Germany

This study is motivated by controversial findings on the change of network connectivity in nanoporous gold (NPG) during coarsening. We used on-lattice kinetic Monte Carlo simulations to study the evolution of size and ligament connectivity of spinodal-like initial microstructures during coarsening. Samples with different solid volume fractions were investigated at temperatures below and above their roughening tran-

sition temperature. Based on the simulation data, we discuss which measure for size is most fundamentally linked to the coarsening kinetics. We also show that the evolution of the connectivity depends sensibly on the solid fraction. This has ramifications for how mechanical properties of nanoporous metals vary when the ligament size is tuned through coarsening.

15 min. break

MM 29.7 Wed 16:45 H46

Fractal abnormal grain growth in nanocrystalline $Pd_{90}Au_{10}$: an experimental study of factors influencing interface fractality — •Christian Braun[1], Raphael Zeller[2], Hanadi Menzel[1], Nils Boussard[1], Jörg Schmauch[1], Carl E. Krill III[2], and Rainer Birringer[1] — [1]Experimental Physics, Saarland University, Germany — [2]Institute of Functional Nanosystems, Ulm University, Germany

With grain sizes below 100 nm, nanocrystalline materials are characterized on the one hand by beneficial properties like high strength, while, on the other hand, being thermally unstable. Grain growth generally starts at temperatures that are significantly lower than usual, and microstructural coarsening often proceeds in an abnormal manner. In nanocrystalline $Pd_{90}Au_{10}$ alloys prepared by inert gas condensation, such abnormal growth leads to an impinged state of grains exhibiting highly irregular shapes, the perimeters of which are characterized by a box-counting fractal dimension of about 1.2.

Insight into the growth mechanism(s) underlying these observations could potentially be gained from a detailed analysis of interface fractality, provided the following questions can be answered: Does self-similarity in grain morphology start from the elementary building blocks of the initial microstructure (i.e., individual nanograins) or at a larger length scale? Does the fractal dimension of an abnormal grain boundary differ between its mobile and impinged states? For both questions, it is important to assess the influence of the Au concentration on abnormal grain nucleation and boundary fractality.

MM 29.8 Wed 17:00 H46

Fractal abnormal grain growth in nanocrystalline $Pd_{90}Au_{10}$: a simulation study comparing possible mechanisms — •Raphael A. Zeller[1], Christian Braun[2], Mingyan Wang[1], Rainer Birringer[2], and Carl E. Krill III[1] — [1]Institute of Functional Nanosystems, Ulm University, Germany — [2]Experimental Physics, Saarland University, Germany

Although the phenomenon of grain growth is well understood in samples having micrometer-sized grains, our understanding of the corresponding process in nanocrystalline materials is rudimentary at best. At the nanoscale the dominant mode of coarsening appears to be abnormal in nature, with a few grains growing orders of magnitude larger than their neighbors. In samples of nanocrystalline $Pd_{90}Au_{10}$ produced by inert gas condensation, microstructural coarsening is doubly abnormal, with the rapidly growing grains observed to send forth "tentacle" into the matrix, quickly encircling nearby grains and then consuming them. The perimeters of the resulting grains resemble those of fractal objects. Recent experiments suggest two possible mechanisms for fractal abnormal grain growth: (1) coalescence via grain rotation or (2) migration according to highly anisotropic grain boundary (reduced) mobilities. By combining a phase field model for boundary migration with boundary-specific selection rules, we have developed a "hybrid" simulation algorithm for each of these scenarios. A comparison of computational results with experiment reveals that one of the proposed mechanisms does a better job than the other at capturing the main features of fractal abnormal grain growth.

MM 29.9 Wed 17:15 H46

Formation and evolution of silver clusters in nanoporous gold: Views from kinetic Monte Carlo simulations — •Bao-Nam Dinh Ngô[1], Yong Li[2], Jürgen Markmann[1,2], and Jörg Weissmüller[1,2] — [1]Institute of Materials Research, Materials Mechanics, Helmholtz-Zentrum Geesthacht, Geesthacht, Germany — [2]Institute of Materials Physics and Technology, Hamburg University of Technology, Hamburg, Germany

Nanoporous gold (NPG) made by dealloying assumes the geometry of an interconnected network of nanoscale ligaments. Thanks to its abundant free surface, the material offers a wide range of potential applications in actuation, catalysis, and sensing. While the fabrication process gives rise to a mostly monolithic network structure, the small remnant of silver content proves to have strong influences on the performance of the material at work. Here, using on-lattice kinetic Monte Carlo simulations, we systematically study the formation and evolution of silver clusters at different stages of dealloying as well as during the subsequent post-dealloying coarsening. We then explore the role of dealloying potentials on the size and number of silver clusters. Finally, we briefly discuss how the environment influences the surface concentration of silver in NPG.

MM 29.10 Wed 17:30 H46

How material structure and properties of Al-Cu Friction Stir Welding seams depend on process parameters — •Roland Marstatt[1], Amanda Zens[2], Sophie Grabmann[2], Michael Friedrich Zaeh[2], and Ferdinand Haider[1] — [1]Chair for Experimental Physics I, University of Augsburg, Universitätsstraße 2, 86159 Augsburg, Germany — [2]Institute for Machine Tools and Industrial Management (iwb), Technical University of Munich, Boltzmannstraße 15, 85748 Garching, Germany

Joining of different metals, for instance of aluminium and copper, is of great industrial interest for applications in light weight construction and electromobility. In contrast to fusion welding techniques, Friction Stir Welding (FSW) does not form a multitude of brittle intermetallic phases upon joint solidification. Only a small layer of intermetallic phases is formed at the interface. Those layers are in the nanometer regime and are the key joining mechanism. In this study, the material structure, especially the intermetallic interface layers, of Al-Cu lap joints are compared with respect to variations of several welding parameters. Therefore aluminium and copper had been welded in temperature controlled mode as well as constant rotational speed mode. The set temperature respectively the set rotational speed had been varied over the possible process window. Also tool velocity, tool length and seam geometry had been varied. The structure of the intermetallic layers, especially its thickness, was measured by electron microscopy and compared to the results of corresponding shear tensile tests with respect to the process variations.

MM 29.11 Wed 17:45 H46

Impact of an Effective Triple-line Energy on the Reactive Wetting of Micro-solder on Miniaturized Cu/Ni Tracks — •Samuel Griffiths and Guido Schmitz — Institute for Materials Science, Stuttgart University, Germany

Micro-solder technologies are ubiquitous in modern electronics industries. Although these industries have driven vast practical advancements in solder technologies, little is understood about the impact of miniaturization on the reflow behaviour of solder on metal conductive tracks.

This work quantifies and analyzes the reflow behavior of Sn-based micro-solder on micro-structured Cu and Ni capillary tracks for various track widths, where solder-reflow experiments have been conducted at 350 °C in a reducing flux environment. We show evidence of an effective triple-line energy (~700 mJ/m) which impacts on the steady state system pressure and quantitatively link the triple-line energy to the solder contact angles. The steady state angle at the solder front is compared with a traditional sessile drop wetting angle measurement and, a systematic deviation is observed and discussed.

A scaling-down of the capillary track size down to a few micrometer leads to the interesting case where the track dimensions approach the size of individual substrate grains, thus requiring a modified model for describing the reflow behavior. Electron microscopic imaging and chemical analysis as well as theoretical modelling based on surface, interface and triple-line energy minimization are applied to support our conclusions.

MM 29.12 Wed 18:00 H46

Aging Processes in an Al(Mg,Si) Alloy Studied by Positron Annihilation Lifetime Spectroscopy — •Laura Resch, Gregor Klinser, Robert Enzinger, Elisabeth Hengge, Wolfgang Sprengel, and Roland Würschum — Institute of Materials Physics, Graz University of Technology, Graz, Austria

Despite the great relevance of age hardenable aluminium alloys, the atomistic processes, i.e. the formation of precipitations, underlying the age hardening are not fully understood yet. Especially, for the investigation of the initial stages and the sequence of precipitation, positron-electron annihilation has been proven to be a very suitable tool. In the present study[1] the specific technique of positron annihilation lifetime spectroscopy is applied to investigate artificial aging of the commercial Al(Mg,Si) alloy (EN-AW6060). On short timescales a

variation of the positron lifetime is observed, which is correlated with a characteristic increase in length, as monitored by in-situ dilatometry. Furthermore, the well-known aging characteristics of the hardness turned out to be directly related to the positron data. The results give hints on the early cluster formation processes. This kind of investigation allows comprehensive analyses of the aging of aluminium alloys within different time scales as well as temperature regimes, which is important for the development of novel, tailored commercial light weight alloys.

[1] L. Resch, G. Klinser, E. Hengge, R. Enzinger, M. Luckabauer, W. Sprengel, R. Würschum, Journal of materials science 53, 14657(2018).