

MM 11: Microstructure and Phase Transformations II - Nucleation/Solidification

Time: Monday 11:30–13:00

Location: IFW B

MM 11.1 Mon 11:30 IFW B

Determination of nucleation rates in a miscibility gap —

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A wetting transition from partial wetting to complete wetting is possible in alloys with a liquid miscibility gap. At a temperature T_p below the critical temperature T_c the alloy separates in a two-phase liquid system which plays an essential role in the microstructure formation of monotectic alloys. One of the liquid phases covers completely the surface of the other phase and the container walls. After the solidification of the first phase, this phase may act as heterogeneous nucleation site for the second phase. We present data of the Bismuth-Gallium system that has a miscibility gap with a critical point at 70 at% Gallium. The aim of the present study is the analysis of the nucleation rate of the crystalline phases with and without a liquid phase separation. We use a self-designed fast scanning calorimeter (up to 10 000 K/s) with nanowatt resolution in order to measure a statistical relevant data set of nucleation temperatures. With the help of the fast scanning calorimeter, we are able to prevent long range phase separation by fast cooling. The nucleation rate is evaluated by a statistical analysis that treats nucleation as a non-homogenous Poisson process.

MM 11.2 Mon 11:45 IFW B

Dependence of accessible undercooling on prior liquid overheating by differential fast scanning calorimeter —

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The dependence of accessible undercooling on prior overheating of a pure tin single micron-sized droplet was studied by differential fast scanning calorimetry (DFSC) with cooling rates from 500 to 10,000 K/s. It is observed experimentally that (i) the degree of undercooling increases first gradually with increasing of prior overheating and reaches then an undercooling plateau; (ii) the accessible undercooling increases initially with increasing cooling rate. However, above a certain cooling rate the accessible undercooling decreases strongly with increasing cooling rate. For overheating levels above that for the onset of the undercooling plateau the undercooling increases with increasing cooling rate. These observed unusual behavior is successfully explained by heterogeneous nucleation in cavities. This mechanism can possibly also describe nucleation in other similar rapid solidification processes.

MM 11.3 Mon 12:00 IFW B

Analysis of the size effect and interfacial structure on the solidification and melting behavior of embedded Bi nanoparticles —

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Abstract Bi nanoparticles embedded in Zn or Al matrices were synthesized by rapid solidification and casting. The kinetics of Bi nanoparticles solidification has been examined by heating and cooling experiments in a differential scanning calorimeter (DSC). Upon heating in the DSC two Bi melting signals occur, one close to the eutectic temperature and the other at a lower temperature. Subsequent cooling leads to two solidification signals. Some Bi particles solidify at a small undercooling with a sharp exothermic peak, whereas other Bi particles solidify at a larger undercooling with a broad exothermic peak. The microstructure was analyzed by transmission electron microscopy (TEM). Cast samples show embedded Bi particles with a size larger than 80 nm and melt spun samples show embedded Bi nanoparticles with a size larger than 5 nm. The Bi nanoparticles that have an elongated and faceted morphology show an orientation relationship with the matrix. The solidification is interpreted using the classical theory of heterogeneous nucleation. Analysis of the results emphasizes the

important role of the local microstructure of embedded Bi nanoparticles and of the liquid*solid interface of embedded Bi nanoparticles with matrix.

MM 11.4 Mon 12:15 IFW B

Dendritic growth of tenfold twins from an undercooled melt of NiZr —

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The solidification of the intermetallic, congruent melting binary alloy Ni50Zr50 is studied in a temperature regime between the melting and the glass transition temperature. This system is well known as a good glass former in a broad concentration range of 30 to 80 at.% Zr. It is studied as a glass forming material, but it has as well a rare behaviour in solidification. We use electrostatic levitation technique to melt and undercool samples with a diameter of 2-3 mm under ultra-high-vacuum conditions. Containerless processing is an effective tool for undercooling metallic melts far below their equilibrium melting temperatures since heterogeneous nucleation on container walls is completely avoided. During crystallisation the rapid increase of the temperature at the solid-liquid interface makes the solidification front visible. The solidification front is recorded by using a high-speed camera with a frame rate of 10.000 (1024x744) frames per second. At high undercoolings the solidification front of Ni50Zr50 shows features which make the investigation of its solidification behaviour particularly interesting. We observed the growth of a solidification front with 10-fold symmetry at a high undercooling temperature of approximately 300 K and a cooling rate of 53 K/s. Funding by Deutsche Forschungsgemeinschaft, under grant HE1601/18.

MM 11.5 Mon 12:30 IFW B

Kristallisationsverhalten und Kompositionsbestimmung von GeSbTe auf Si(111) mittels in-situ Transmissionselektronenmikroskopie und Elektronenenergieverlustspektroskopie —

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Die Funktionalität von Phasenwechselmaterialien wie Ge-Sb-Te-Verbindungen (GST) basiert auf einer schnellen und reversiblen Phasenumwandlung zwischen amorphem und kristallinem Zustand. An dieser Stelle wird zunächst der amorphe Ausgangszustand strukturell analysiert und beschrieben. Anschließend wird der Einfluss eines einkristallinen Si-Substrats auf das Kristallisationsverhalten mittels in-situ Transmissionselektronenmikroskopie (TEM) im Temperaturbereich von Raumtemperatur bis 600°C untersucht. Im Bereich der amorph-kristallinen Übergangstemperatur (120-140°C) wird ein homogenes Keimbildungsverhalten mit einem anschließenden lateralen Kornwachstum beobachtet. Oberhalb der zweiten Übergangstemperatur (400-600°C) konnte erstmals eine Umorientierung und Ausrichtung der Kristallstruktur bezüglich des Substartes beobachtet werden. Es werden zwei Mechanismen zur Ausbildung dieser Orientierung diskutiert. Für die eingehende Untersuchung ist eine exakte Bestimmung der chemischen Komposition insbesondere in Hinblick auf die Homogenität auf der Nanometer-Skala entscheidend. Die Bestimmung der chemischen Zusammensetzung des tertiären GST erfolgte mittels Elektronenenergieverlustspektroskopie (EELS) sowohl am amorphen Ausgangszustand wie auch am kristallinen Endzustand.

MM 11.6 Mon 12:45 IFW B

Dendritic growth kinetics of undercooled Fe-B alloy melts —

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In metallic alloys dendritic growth is the major crystal growth mode controlling the evolution of the microstructure during solidification. The present work investigates the growth kinetics on pure Fe and Fe-B alloy melts in order to verify models for dendritic growth in undercooled melts. In detail, we analyze the effect of solute redistribution and solute trapping on the rapid growth of dilute Fe-1 at.% B alloy with B showing a small partitioning coefficient. For in-situ studies on

deeply undercooled melts electromagnetic levitation technique (EML) is used. The rapid solidification is recorded by a high-speed camera. Undercoolings from 50 K to 300 K prior to solidification leads to growth velocities of several cm/s up to m/s. Concerning terrestrial EML the electromagnetic field necessary for levitation induces strong convective fluid flow inside the melt. To investigate the effect of convection on the

growth kinetics experiments on parabolic flight are carried out under reduced gravity conditions. The microstructure of the solidified sample is analyzed by SEM and EBSD. Experimental results will be presented and discussed in the frame of current models for dendritic growth in undercooled melts. This research work is supported by DFG contract HE1601/18 and by ESA under contract 4200020277 and 4200014980.