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Influence of time-variant temperature gradients on resolidifying mushy zones

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ABSTRACT

The influence of thermal diffusion in a stationary temperature gradient on the resolidification of a mushy zone is investigated. The temperature distribution in the sample is calculated by solving the heat diffusion equation numerically for the varying phase fractions, aiming at predicting the position of the liquid and solidus temperatures depending on the thermal diffusivities (heat conductivities and heat capacities C_p) in the different phases. Phase fractions and C_p values for thermal diffusivities are calculated using the thermochemical library ChemApp. Experimental investigations are performed using eutectic and peritectic alloys in the system Al–Cu. It is found that the solidus and peritectic isotherms migrate considerably during annealing. Micrographs of annealed samples show a more or less pronounced transition zone at the positions of the solidus and peritectic temperatures. The extension of the transition zone corresponds reasonably well with the experimentally determined migration distance of the solidus and peritectic isotherms.

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1. Introduction

Exposing a cylindrical sample to a temperature gradient by heating one side and cooling the other one causes partial melting of the sample between the positions of the solidus and liquidus temperatures. At initial stages, the local fractions of solid and liquid are (approximately) the ones that can be derived from the phase diagram using the lever rule. Local equilibration at the interfaces along the sample generates continually changing equilibrium concentrations at the solid/liquid interfaces, leading to a macroscopic concentration gradient in both the liquid and solid phases. This in turn stimulates mass transport towards higher temperatures. With increasing annealing time in the temperature gradient, the local fractions of solid and liquid change, the mushy zone resolidifies and eventually is fully solid with a plane front at the position of the liquidus temperature. The concentrations along the completely resolidified sample follow the solidus concentrations [1]. Thus, single phase regions of the primary phase and, in peritectic alloys, the secondary phase(s) will form after a sufficiently long holding time [2,3].

In published work on mushy zone resolidification, it is mostly implicitly assumed that the temperature gradient does not vary with time, which for a *post-mortem* characterization [4] or a (semi-quantitative) *in-situ* evaluation of local processes [5] is certainly sufficient. The assumption of a constant gradient is generally not discussed in detail, even though it is sometimes mentioned that the extension of the mushy zone and the temperature gradient may vary over time, resulting in a difference of liquid droplet migration velocities at the beginning and at the end of the stabilization stage [6]. Constant temperature gradients over the mushy zone that do not change with time were also used in the few instances where the processes occurring during resolidification of the mushy zone are modeled [4,7]. However, a rather straightforward reasoning shows that the temperature gradient cannot be constant during mushy zone resolidification. The solid and liquid phases have different thermal conductivities, consequently at a given heat flux through each section of the sample, the temperature gradients must adjust according to the thermal conductivities as the local phase fractions change. This implies that also the position of the isotherms will change during the experiment, which is experimentally directly observable if at a specific isotherm a change in microstructure occurs, as e.g. at a peritectic or eutectic temperature.

In the present work, the influence of the varying temperature gradient on mushy zone resolidification is investigated. The temperature distributions along the mushy zone prior to and after its resolidification are simulated by solving the thermal diffusion equation. For the determination of the local solid and liquid phase fractions, the thermochemical library ChemApp was used that is

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based on the Calphad method. The calculated temperature profiles that predict the migration of the solidus, liquidus and peritectic temperature in Al–Cu alloys are compared with microstructures of an Al–4 wt%Cu alloy and a Cu–40 wt%Al alloy that were held in a temperature gradient until the resolidification process was finished.

2. Experimental

2.1. Mushy zone resolidification

Temperature gradient annealing experiments were carried out using two alloys of the Al–Cu system: an Al–4 wt%Cu alloy that resolidifies as single phase to an aluminum solid solution [8], and a peritectic Cu–40 wt%Al alloy that resolidifies to the intermetallic phases η and ε in spatially separated regions of the sample [3].

Rods of 8 mm diameter of the two alloys were cast with a cooling rate of approximately 10 K/s from the pure elements with $>99.9\%$ purity, and pieces of 8 cm length were cut. They were then placed in an alumina tube and melted at the upper end using a high frequency induction furnace, while the lower end was cooled by streaming water. For temperature control, a thermocouple was attached to the sample slightly below the heating coil of the induction furnace. The control temperature was 50 K below the solidus temperature that was taken from the phase diagram and confirmed by calculation using ChemApp and the SGTE2011 data base. The average heating rate at the position of the pyrometer was 2 K/s until the final control temperature was reached. With the given set-up, mean temperature gradients in the mush of 26 K/mm (Al–4 wt%Cu) and 50 K/mm (Cu–40 wt%Al) were achieved in the mushy zone. The mean temperature gradients were determined by dividing the solidification interval from the phase diagram by the length of the mushy zone (length between quenched liquid and fully solid structure). The curvature of the interfaces at solidus and liquidus was neglected for determining temperature gradients from the microstructure, as both isotherms are concave and the slight differences in curvature would only exert a minor influence on the temperature gradient. After annealing times of 15 min, 30 min and 3 h, respectively, the samples were quenched by turning off the heating. For microstructural analysis the samples were ground with SiC paper (80–4000 grain) and polished with diamond spray (0.25 μm) before the microstructural investigations using the Scanning Electron Microscope (SEM). The initial concentrations and phase compositions were determined by Energy Dispersive X-ray Spectroscopy (EDX).

2.2. Determination of the thermal diffusivity of the ε phase

Since no literature values for the thermal diffusivity of the ε phase were found, its value was determined experimentally. A stationary temperature gradient was generated in the Cu–40 wt% Al sample that contained spatially separated ε and η phases (see Fig. 1) generated in the previous mushy zone resolidification experiment. The different thermal diffusivities in the as-cast microstructure, η and ε and quenched liquid regions lead to different local temperature gradients in those regions. The temperature gradient in regions with low thermal diffusivity will be steeper than in regions with high thermal diffusivity. Since, after an initial transient, the thermal flux across all sections of the sample must be equal, the product of local thermal gradient G_T and local thermal diffusivity α is equal as well. The thermal diffusivity of ε is thus straightforwardly defined by:

$$G_{T,\varepsilon}\alpha_\varepsilon = G_{T,\eta}\alpha_\eta \quad (1)$$

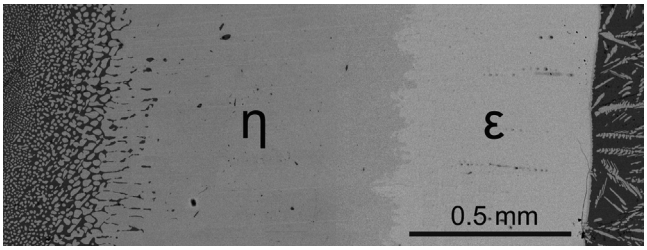

Fig. 1. BSE image of a Cu–40 wt%Al sample that was held in a temperature gradient for 3 h. Spatially separated sections of ε and η phase, respectively, have formed along the sample.

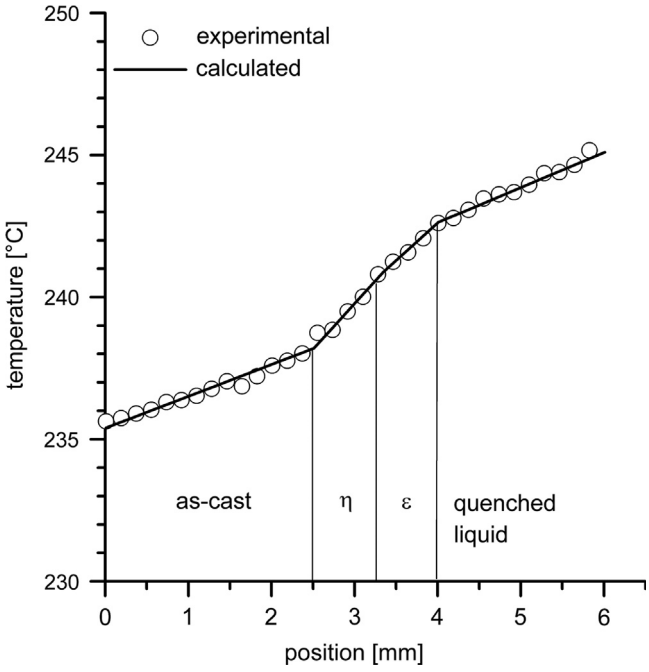

Fig. 2. Measured and calculated temperature profiles in a Cu–40 wt%Al sample; the calculated curve was obtained by starting with an estimated value for the thermal diffusivity of the ε phase and using its value for fitting the resulting curve to the measured profile.

The temperature profile along the sample was recorded using an infrared line camera with 512 pixel. The ground and polished side of the sample was covered by a thin layer of BN to prevent artefacts due to varying optical properties of the different intermetallic phases. To calibrate the infrared line camera, a thermocouple was positioned inside the sample, and the intensity that the infrared line camera measured at the position of the thermocouple was recorded for different temperatures by the thermocouple. A steady temperature distribution was obtained by heating one end of the sample to a temperature of 400 °C using induction heating and temperature control by a pyrometer while the other end of the sample was air cooled.

The resulting temperature profile is shown in Fig. 2. The former mushy zone consists of two regions, particularly those of the ε and η phases, respectively. The temperature gradient in the intermetallic phases is significantly steeper than the gradients in the as-cast microstructure and the quenched liquid, respectively. The thermal diffusivity in ε and η is thus lower than in the as-cast microstructure and the quenched liquid that both contain a high fraction of θ-Al_2Cu phase ($>60\%$). The temperature gradients in the ε and η phases only differ slightly. While the error in absolute temperatures in the temperature profile may be considerable (<5 K), the error in the temperature gradients is assumed to be small, since relative

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