# Solidification Paths of Al-Alloys

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#### Abstract

The solidification curves of Al-rich binary Al-Si and ternary Al-Si-Cu alloys have been investigated using two experimental methods. The samples were directionally solidified and quenched rapidly with a liquid metal cooling. In addition, the materials were thermally analysed with DSC under the same cooling rates as in the previous experiments. Apart from these two experimental methods, theoretical analyses have been carried out using the lever rule and the Scheil model.

Results of the two experimental methods are compared to each other and to the theoretical predictions. The significant differences found in the solidification curves, mainly in the beginning of the process, are critically discussed.

Keywords: directional solidification, mushy zone, solid fraction, Al alloy

### 1. Introduction

Understanding the formation of microstructure especially of multicomponent alloys is crucial for the command of solidification processes. For controlled development and optimization of castings, numerical simulation becomes more and more significant nowadays. In simulating the evolution of the structure in a micromodel the build-up of solid fraction,  $f_s$ , within the mushy zone is an important parameter. As only little experimental data is available, in simulations the progress of  $f_s$  is often calculated using very simple models. To overcome these limitations the aim of this work is to provide an experimental analysis of the evolution of solid fraction.

A binary Al-4.8wt%Si alloy and a ternary Al-5.5wt%Si-1.5wt%Cu alloy were studied using quenching experiments as well as DSC measurements.

## 2. Experimental Study

For the directional soldification experiments (DS) a modified Bridgman furnace was used. The process conditions are described in detail in [1, 2]. The cylindrical samples, 8 mm in diameter, were directionally solidified with a constant withdrawal velocity of  $0.5 \, \mathrm{mm/min}$ . The temperature gradient was determined to be  $16 \, \mathrm{K/mm}$ . After withdrawing a length of the sample sufficient to reach steady state conditions, the samples were rapidly quenched with a GaIn liquid metal cooling. To determine the evolution of solid fraction as a function of temperature, a series of cross sections within the mushy zone of the directionally solidified samples similar to the one in figure 1 were studied by image analysis. As the structure is strongly branched and many seemingly separated segments of  $\alpha$ -phase are visible in the photograph, adapted filters were used for the image analysis [1].

The phase transformations during solidification involve thermal effects which give information about the progress of the solidification process. For thermal analysis of the alloys Al-4.8wt%Si and Al-5.5wt%Si-1.5wt%Cu (weight of samples taken from a bulk material  $\approx 5$  mg) DSC measurements with cooling rates of 10 and 15 K/min respectively were used. From the cooling curves measured by DSC, the solid fraction could directly

were used. From the cooling curves measured by DSC, the solid fraction could directly be estimated by determining the transformed volume fraction at a certain temperature which is proportional to the amount of heat released at this temperature [3].

With the  $f_s$  values of these two independent experimental methods a critical evaluation of the measurement and analysis processes was possible.

For theoretical calculations, the differential forms of the lever rule and the Scheil model were combined with a commercial software for thermodynamic calculations (ChemSage) [4]. In this way  $f_s$  could be determined taking the temperature dependence of the distribution coefficient into account.

## 3. Results and discussions

During quenching of the directionally solidified samples the initial solid-liquid interface became unstable and cells had grown onto the dendrite branches of the primary structure, as shown in figure 2. Until the interface became unstable an additive amount of solid fraction had built up which should not be counted to the amount of primary phase. Up to now only rough estimation of this quenching effect has been possible. For a numerical determination of this effect a stability analysis of an accelerated growing dendritic interface would be needed (compare [5]). Thus, besides statistical fluctuations, the quenching effect gives a significant, but difficult to estimate uncertainty in the measured value of the solid fraction.

In figure 3 the experimentally determined solid fractions for both alloys are compared with the predictions of the lever rule and the Scheil model [6]. The solid fractions measured from quenching experiments are always higher than those found from DSC. Especially at the beginning of the mushy zone, the solid fractions from the directional solidification increase much steeper than the  $f_s$  values from DSC. These results of the evolution of solid fraction are supported through studies of the morphology. Longitudinal sections of directionally solidified samples and microscope studies of decanted microstructures suggest a strong increase of solid fraction within a zone of free growth [1]. This zone of free growth comprehends the region from the dendrite tips to the point where overlapping diffusion fields of neighboring dendrites inhibit further growth. After this

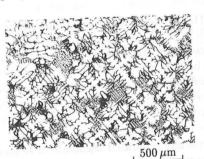


Figure 1: Cross section of Al-4.8wt%Si showing the typical primary dendrite structure with a lot of seemingly seperated regions of  $\alpha$ -phase.

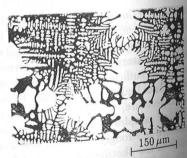


Figure 2: Primary dendrites with instable solid/liquid-interface due to quenching.

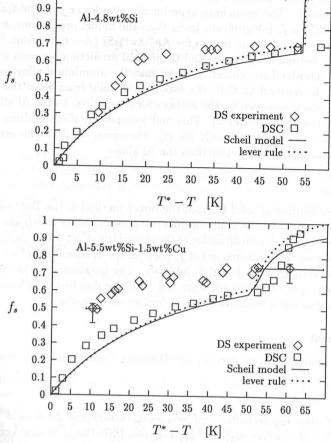


Figure 3: Solid fraction of Al-4.8wt%Si and Al-5.5wt%Si-1.5wt%Cu as function of the temperature difference to the tip temperature  $T^*$ .  $(v(DS) = 0.5 \, \text{mm/min}$ ,  $\dot{T}(DSC) = 15 \, \text{K/min})$ 

zone solid fraction enhances more slowly.

The model calculations yield a slower increase than both experimentally determined curves. In particular, the rapid increase of the measured solid fraction at the beginning is not reproduced (compare also [7]). In addition, the calculated curves differ from the experimentally determined curves at the starting point of eutectic precipitation. For Al-4.8wt%Si the eutectic temperature determined from DSC is about 5 K lower than that given from the phase diagram. This may be traceable to inhomogeneities in the material due to the small size of the samples. With an uncertainty of about ±8 K near the eutectic temperature the values of the quenching experiments are not well defined. On the other hand, especially for ternary systems, the temperatures given from thermodynamical The means are not very accurate.

The measuring errors of the experimental results are greater than the range determined by the limiting cases of equilibrium (lever rule) and non-equilibrium (Scheil model).

Al-4.8wt%Si, 36% for Al-5.5wt%Si-1.5wt%Cu) lie inbetween the predictions of lever rule and Scheil model. The quenching experiments give lower eutectic fractions. For Al-5.5wt%Si-1.5wt%Cu f<sub>e</sub> is significant lower than the model predictions, even those of the lever rule. Together with the results for Al-7.8wt%Si ( $f_e$ = 62%) from [2] it follows that the difference between experiment and theoretical prediction becomes stronger with rising amount of interdendritic eutectic. Note, that the aluminium alloys solidify in at least two phases. In contrast to that, the evolution of solid fraction of the Ni-base superalloy SRR 99, which was used by the authors for comparison to the Al alloys in [2], is well described by the Scheil model [1]. This multicomponent alloy solidifies quasi single phase with a amount of eutectic of only 3% [8]. Therefore, it fulfills the assumptions of the theoretical modelling much better than the Al alloys.

## 4. Conclusions

In analysing the evolution of solid fraction the direct method of the DSC measurements shows clear advantages over the study of quenching experiments which are subject to a high effort of preparation. The estimation of the quenching effect which currently imposes a large uncertainty on the measurement of  $f_s$  requires the development of a stability analysis for an accelerated growing dendritic interface. The question, whether the difference in the results from quenching experiments and DSC are due to the differences between directional and equiaxed solidification, needs further investigation.

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