Directional Solidification of AlSi Alloys with Fe Intermetallic Phases

P. Mikołajczak [a, b, *], L. Ratke [b]

[a] Institute of Materials Technology, Poznan University of Technology, Piotrowo 3, 60-965 Poznan, Poland
[b] Institut für Materialphysik im Weltraum, German Aerospace Center DLR,
Linder Höhe, 51147 Köln, Germany
*Corresponding author. E-mail address: Piotr.Mikolajczak@put.poznan.pl

Abstract

Directional solidification technique is an important research instrument to study solidification of metals and alloys. In the paper the model [6,7,8] of directional solidification in special Artemis-3 facility was presented. The current work aimed to propose the ease and efficient way in calibrating the facility. The introduced M coefficient allowed effective calibration and implementation of defined thermal conditions. The specimens of AlSi alloys with Fe-rich intermetallics and especially deleterious β-Al$_5$FeSi were processed by controlled solidification velocity, temperature gradient and cooling rate.

Keywords: Directional solidification, Aluminum alloys, Calibration, Fe-rich intermetallic phases, β-Al$_5$FeSi

1. Introduction

Directional solidification technology enables formation of materials during directional transition from liquid to solid state along direction specified trough thermal gradient [1]. As first directional solidification was applied to prepare turbine blades, and can be used to improve functional and structural characteristics of materials, such as single crystal super alloys, high temperature intermetallic compounds, in-situ eutectic composites. Directional solidification technique is also an important research instrument to study solidification theory of metals and alloys, because it helps to achieve controllable cooling rate in a broad range, solidification structure from near-equilibrium to far-from equilibrium, interface evolution, solute redistribution, phase selection, crystal growth instability [2,3]. From directional solidification well known are instruments of Czochralski [4], Bridgman-Stockbarger [5] and Chalmers. One of the methods for conduction of directional solidification is the aerogel based power down method that uses two controlled heaters [6,7,8]. The method applies control of each heater independently introducing in the specimen the projected temperature gradient, cooling rate and velocity of the solidification front. Application of the aerogel crucible allows to control the position of solidification front in time (because of its transparency) and extremely low thermal conductivity maintain plane solidification front in processed specimens.

Aluminum alloys are characterized by good application characteristics and are replacing traditional materials in many areas e.g., aerospace and machinery. One of the impurities in AlSi alloys is iron causing formation of Fe-rich intermetallics, with especially detrimental β-Al$_5$FeSi phases.

In the current paper we have concentrated on the calibration method of Artemis [6,8] to control temperature and we proposed factor *M*, by solidification of AlSi alloys with Fe-rich intermetallics.

2. Methodology

This study comprises calibration of Artemis [6,7,8] special facility, that is Bridgman-like furnace for directional solidification. In the centre of Artemis two heating elements are located with a cylindrical specimen (8 mm diameter and 120 length) between (Fig. 1). The specimen is surrounded by a piece of silica aerogel as crucible and thermal insulator [6,7]. Bottom heater was equipped with a cylindrical cooling element attached to cooling system (copper plate cooled with water). The heat flows between heaters trough specimen and leaves them trough cooling system. Thanks to construction and vacuum only small part of the heat radiate to facility chamber [7,8]. The aerogel crucible with excellent insulating properties secure almost flat isotherm in the specimens and directional solidification. The heaters are controlled with PID unit allowing solidification with supposed constant velocity, temperature gradient and cooling rate [8,9]. Because the silica aerogel is transparent, it is possible to record the brightness intensity with special CCD camera. Analysing brightness course allows to determine the solidification front in time and solidification velocity.

The experiments with AlSi alloys were conducted by temperature gradient G=3 K/mm and solidification velocity v=0.04 mm/s with and without fluid flow induced inside the specimen by a rotating magnetic field (RMF) with 6 mT.
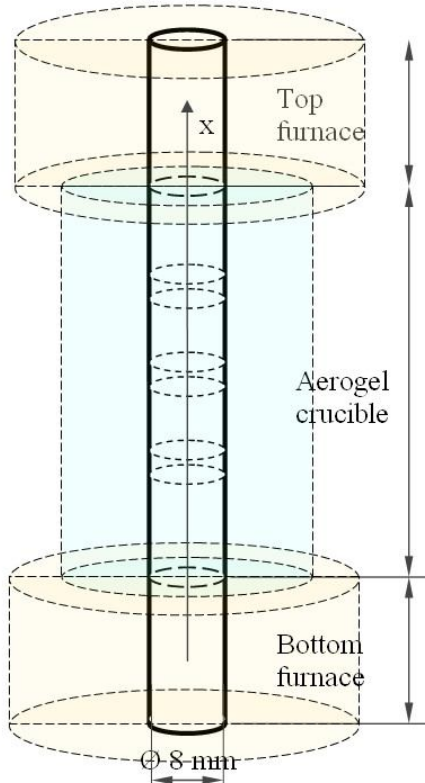

Fig. 1. Schematic picture of the Artemis-3 facility. The specimen with 8 mm diameter mounted in the aerogel crucible (light blue) between bottom and top furnace (light yellow).The X axis located in the specimen center

3. Results and Discussion

Artemis allows for solidification according to planed thermal conditions thanks to fully controlled top and bottom furnaces and aerogel crucible. For solidification with expected thermal gradient, velocity and cooling rate, in the heaters we need to assume temperature profile in time. Ratke [6] described the thermal processes in Artemis with help of Stefan condition:

$$\Delta H_V \frac{dy}{dt} = \lambda_S \frac{\partial T^S}{\partial x}\bigg|_y - \lambda_L \frac{\partial T^L}{\partial x}\bigg|_y \tag{1}$$

where λ_L λ_S are the thermal conductivity in the liquid and solid, ΔH_V is the enthalpy of fusion per volume and $T^{L,S}$ are the temperatures in the liquid and solid, $dy/dt=v$ is solidification velocity. For ease of the model both gradients in liquid G_L and solid G_S are taken [6] as approximately linear, then:

$$\frac{\partial T^L}{\partial x}\bigg|_y = \frac{T_{top} - T_m}{L - y} = G_L \tag{2a}$$

$$\frac{\partial T^S}{\partial x}\bigg|_y = \frac{T_m - T_{bot}}{y} = G_S \tag{2b}$$

where T_{top} and T_{bot} are temperatures in the top and bottom furnace, T_m is the melting temperature, y position of solidification front and L the effective gradient length. After some reconstruction [8] the Eq. (1) become:

$$\rho \cdot \Delta H \cdot v + \lambda_L \cdot G_L = \lambda_S \cdot G_S \tag{3}$$

and calculated on G_S [8] is:

$$G_S = \beta + \alpha \cdot G_L \tag{4}$$

where

$$\alpha = \frac{\lambda_L}{\lambda_S} \qquad \text{and} \qquad \beta = \frac{\rho \cdot \Delta H \cdot v}{\lambda_S} \tag{5}$$

with ρ density at solid-liquid transition, ΔH the enthalpy of fusion and v solidification velocity. Next reconstructions according to [8] give temperature in top T_{top}(t) and bottom T_{bot}(t):

$$T_{top}(t) = T_m + G_L(L - v \cdot t) \tag{6}$$

$$T_{bot}(t) = T_m - (\beta + G_L \cdot \alpha)v \cdot t \tag{7}$$

$$T_{bot}(t) = T_m - \left(\frac{\rho \Delta H v}{\lambda_S} + G_L \cdot \frac{\lambda_L}{\lambda_S}\right)v \cdot t \tag{8}$$

For the determination of the proper temperature profile it is necessary to know thermal properties of processed alloy ρ, ΔH, λ_L, λ_S and the effective gradient length L. Thermal properties of the

studied alloys and value L reveal uncertainty coming from the experimental measurements, but especially from the variety of the alloys studied and access to such data. Steinbach [8] has proposed the determination of L trough iterative experiments. By supposed initial velocity v_{ini}=0.06 (mm/s) and temperature gradient G=3 (K/mm) from the conducted experiment the real solidification velocity v_{real} was gained. Thanks some similar experiments and the data of many velocities v_{real}, the extrapolation function can be calculated and final proper velocity v_{ini} may be determined. In the study [8] has also been proposed the methodology and equations for real gradient determination in the processed specimens. Ratke [9] has proposed the formulas for bottom heater cooling rate dT_{bot}/dt, but it requires calculation of the coefficients (a and b) describing the influence of alloys thermal properties and L, and all have to be determined experimentally. The methodology [9] of a and b determination require two experiments with various cooling rates T_{bot1} and T_{bot2} in bottom furnace, and received two real solidification velocities v_1 and v_2 are used in mathematical formulas for a and b calculation. In the methodology for L it is necessary do conduct two solidification processes with stopping the solidification front at specified position in the specimen between both furnaces and with two various temperatures of the process stopping.

Since Artemis allows free control of the temperatures in both furnaces, it is also possible the solidification with constant acceleration [6]. It is similar to real casting where the solidification varies in time and across the casting piece. Such solidification by varying velocity and constant gradient require additional data that can be determined to the above mentioned methodology for solidification with constant velocity [6].

Solidification experiments for the AlSi alloys with Fe-rich intermetallics solidifying with the same [10,11] or various solidification velocities v required precise determination of v_{ini}. In the study we propose the methodology that need three or four experiments for proper process parameters determination. Literature data and alloy data bases in casting solidification software showed that alloys physical properties ρ, ΔH, λ_L, λ_S reveal uncertainty. The determination of correct values each mentioned properties needed experimental procedure in Artemis for each of them. To prevent many experiments we proposed some constant values of ρ, ΔH, λ_L, λ_S and introduced the coefficient M, that as only one was modified and changed the value of the right hand side of Eq. (8) modifying it to Eq. (9).

$$T_{bot}(t) = T_m - \left(\frac{\rho \Delta H v}{\lambda_S} + G_L \cdot \frac{\lambda_L}{\lambda_S} \right) v \cdot t \cdot M \qquad (9)$$

In his way it was possible to conduct only two experiments, or by higher accuracy three or four experiments. The coefficient M initially assumed as M=1 was changed to M=0.9 and M=1.1 for experiments giving real solidification velocities v_{real}, next trough the extrapolation for M=0.9/1.0/1.1 and v_{real1}, v_{real2}, v_{real3}, we calculated the most proper value of M and conducted the correct experiments.

The measurement of solidification velocity was performed as the measurement of intensity [6] in functions of time of the specimen surface trough aerogel crucible. The measured intensity curves with a peak pointing on solidification front are well visible on Fig. 2. Fig. 3 presents the gained real solidification velocities in function of M coefficient and the extrapolation function for fitting the best M value. Analogical methodology was applied for L.
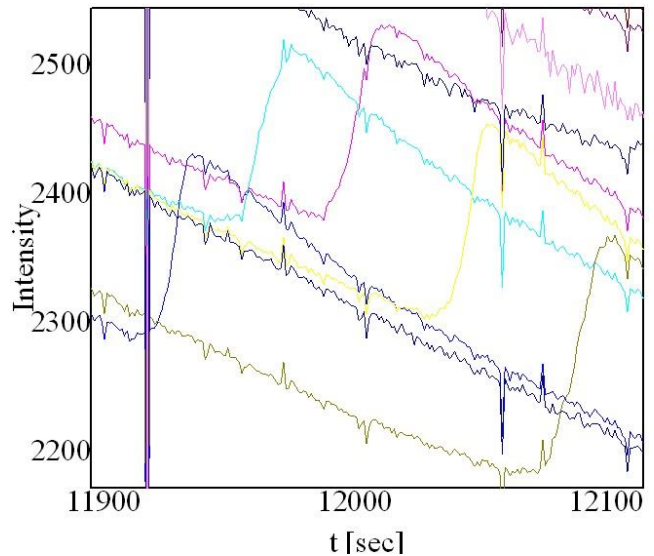

Fig. 2. Intensity in function of time recorder from the specimen sample trough the transparent aerogel crucible with help of the CCD-camera. Solidification of Al-5 wt.% Si-0.5 wt.% Fe alloy, velocity v=0.04 mm/s, gradient G=3 K/mm
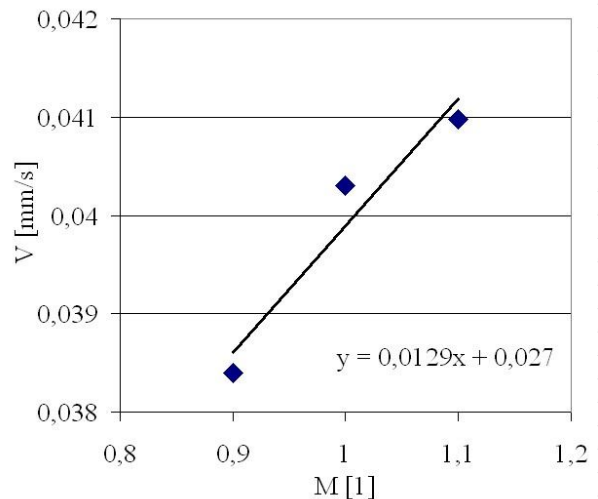

Fig. 3. The example for solidification velocity v=0.04 mm/s of the gained relationship between M coefficient and v_{real}. Solidification of Al-5 wt.% Si-0.5 wt.% Fe alloy

The experiments allowed for precise solidification of AlSi alloys with iron rich intermetallics without and with fluid flow generated in the specimens. The applied methodology secured the solidification with planned front velocity, temperature gradient and cooling rate in the specimens. The gained figures (Fig. 4 a and b) show the microstructures of AlSi alloys solidified with presence of β-Al$_5$FeSi needles.
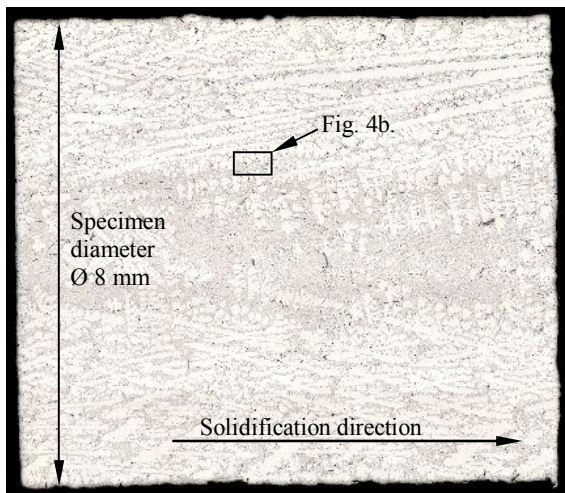
Fig. 4a. A microstructure on the longitudinal section of the specimen of Al-7wt.%Si-1.0wt%Fe alloy solidified by fluid flow. Solidification velocity v=0.04 mm/s, G=3 K/mm
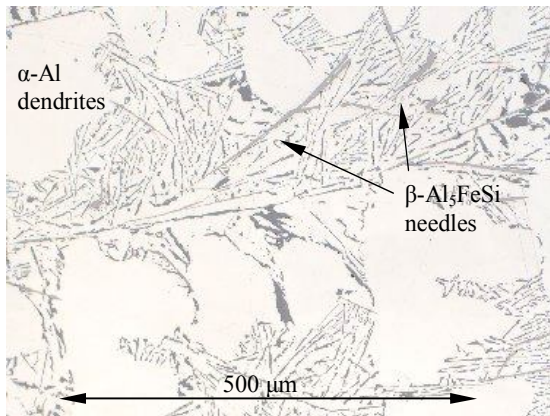

Fig. 4b. Zoomed detail from Fig 4a. with well visible β-Al$_5$FeSi needles (light grey) between α-Al dendrites (white) in Al-7wt.%Si-1.0wt%Fe alloy

4. Conclusions

Directional solidification offers good possibilities to study the microstructure formed by controlled solidification conditions.

In the paper we proposed the methodology for calibration of directional solidification facility. The introduced coefficient M substituted prospecting the most suitable physical parameters considered in the model describing solidification in Artemis. Using M coefficient shortens the iterative experiments routine and can be an effective way leading to studies on the influence of solidification conditions in the microstructure.

The experiments calibrated with introduced M coefficient allowed for precisely controlled solidification conditions of AlSi alloys with iron rich intermetallics.

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