THERMAL AND METALLOGRAPHIC PARAMETERS EVOLUTION DURING SOLIDIFICATION OF Zn-Sn ALLOYS

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Abstract

In the present research the horizontal directional solidification Zn-Sn alloys (Zn-4wt.%Sn, Zn-6wt.%Sn, Zn-10wt.%Sn and Zn-30wt.%Sn) was carried out by extracting heat in two opposite directions. For this purpose, ceramic molds, a muffle, a horizontal furnace with two heat extraction systems at the ends, a measurement, acquisition and recording temperature system, eight K-type thermocouples and an electronic recorder were used. The main parameters involved in the columnar-to-equiaxed transition, CET, were: the moment of start and end of solidification at each position under consideration, cooling rates, the average temperature gradients and critical temperature gradients at the CET, the average speed of liquid and solid interphases left and right, advancing in opposite directions. Also, solidification kinetics was found to be different between horizontal solidification two-way heat extractions, with preliminary results of the vertical unidirectional solidification. In the solidification experiments, the velocity and accelerations of the liquidus and solidus interphases usually occurs at the end solidification zone of the samples.

Introduction

Solidification of metal alloys, which starts in the external equiaxed region (chill), results in two basic types of structures in a single solidified alloy sample: columnar and equiaxed. The presence of both structures indicates the occurrence of the columnar-to-equiaxed transition phenomenon [1].

Studying the columnar-to-equiaxed transition, CET, is of great technological interest for the evaluation and design of the mechanical properties of the solidification products. To this end it is necessary to understand the mechanisms by which it occurs. As found in previous studies [2-5], the CET occurs by competition between columnar and equiaxed growth. Mainly is controlled by the parameters of casting, such as the alloy composition, the density of the present nuclei in the liquid, the cooling capacity of the metal / mold, and the degree of convection in the liquid [7-10].

The aim of the present work is to solidify horizontally and directionally Zn-Sn alloys of different compositions (Zn-4wt.%Sn, Zn-6wt.%Sn, Zn-10wt.%Sn and Zn-30wt.%Sn), and analyze the evolution of the thermal parameters with the structures.

Materials and Methods

The casting assembly used in solidification experiments has been detailed in a previous article [7-10]. Thus, Zn-4wt.%Sn, Zn-6wt.%Sn, Zn-10wt.%Sn and Zn-30wt.%Sn alloy samples were horizontally solidified. Initially the melt was allowed to reach the selected temperature and then, the furnace power was turned off and the melt was allowed to solidify from the bottom. Ceramic molds of 50 mm in diameter were used for horizontal solidification experiments cooled from both ends. Eight made K-type thermocouples were used in the experimental setup. For the horizontal setup, thermocouples were fabricated with thin chromel-alumel wires of 0.5 mm diameter that were inserted into bifilar ceramics of ~ 4.0 mm external diameter and ~ 1.0 mm hollow diameter and introduced inside Pyrex® glass rods of 7.0 mm external diameter and ~ 5.3 mm internal diameter. Adjacent thermocouples were located at a distance of ~ 20 mm. Temperatures were measured at regular intervals of 10 seconds.

A picture of the horizontal experimental device is shown in Figure 1. Small 140 mm long hemicylindrical probes of Zn-Sn alloy were solidified in the horizontal setup. The heat flux toward the ends of the sample was obtained by two cooling systems located at the ends of the ceramic crucible. In this setup, temperatures at eight different positions were measured using a TC 7003C acquisition system and recorded using SensorWatch® software every 1 minute in a compatible PC from the early beginning until the end of solidification. Alloys were prepared with high purity metals (electrolytic zinc and commercial grade tin). For the horizontal setup, a set of five specimens of each alloy concentration were prepared. The alloy was first melted and mixed in a graphite crucible using a conventional furnace and then poured into a previously heated ceramic crucible. The crucible with the alloy was located into the horizontal furnace and heated up above the melting point of the alloy. The solidification of the sample was obtained by cooling down the alloy using the cooling system which extracts the heat toward both ends.

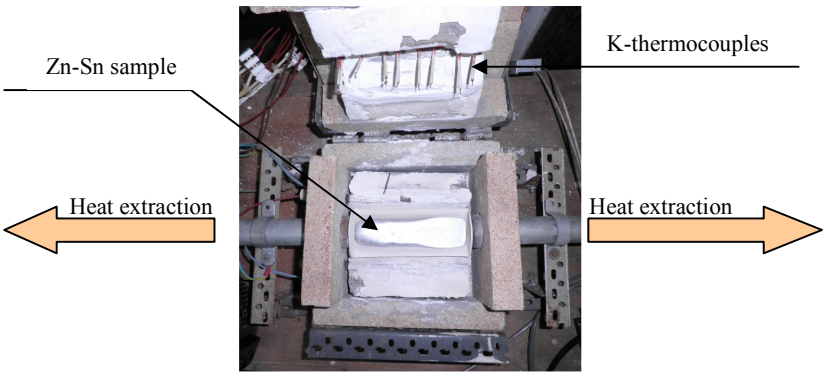


Figure 1. Details of the horizontal experimental device.

After solidification the samples were cut in the longitudinal direction, polished with emery paper and etched to reveal the structure. The reagent used was a solution of HCl acid (70%) during 120 seconds [10]. Typical resulting macrographs can be seen in Figure 2 for Zn-4wt.%Sn, Zn-6wt.%Al, Zn-10wt.%Al and Zn-30wt.%Sn alloys.

To reveal the microstructure a solution containing 5 g CrO_3, 0.5 g Na_2SO_4 and 100 ml H_2O (Palmerston´s reagent) was used. The etching time varied from 15 to 20 seconds, depending on the alloy solute content. After etching, the samples were rinsed in a solution of 20 g CrO_3 and 100 ml H_2O before optical microscopy examination to observe the position of the CET [10].

(a) (b) (c) (d)

Figure 2. Macrographs corresponding to: (a) Zn-4wt.%Sn, (b) Zn-6wt.%Sn, (c) Zn-10wt.%Sn and (d) Zn-30wt.%Sn alloys.

Results and Discussion

The temperature profiles were determined from the measurements during solidification at the different thermocouple positions. The temperature versus time curves for one experiment corresponding to Zn-4wt.%Sn, Zn-6wt.%Al, Zn-10wt.%Al and Zn-30wt.%Sn alloys concentrations are presented in Figure 3. The thermocouple T_1 and T_8 are the first to reach the solidification fronts. From the temperatures versus time graphs it is possible to calculate the cooling velocity in the melt, $\dot{T}$. The velocity associated to each experiment is the average value of the slopes determined from the graphs. The start and the end of solidification at each thermocouple determine the positions of the solidification fronts versus time, which correspond to the liquidus and solidus temperatures, respectively. Both points are detected by the changes in the slopes of the cooling curves at the start and end of solidification. The local solidification time at each thermocouple location is determined by the period of time taken for the temperature to go from the liquidus temperature to the solidus temperature. The velocity of the liquidus solidification fronts was calculated as the distance between thermocouples divided by the time taken by liquidus temperature moves from the lower to the upper thermocouple. These velocities were named as V_L for the liquidus velocity and V_S for the solidus velocity. The liquid thermal gradient, G_L, at all times was calculated straightforward, dividing the temperature difference between two thermocouples by the separation distance between them.

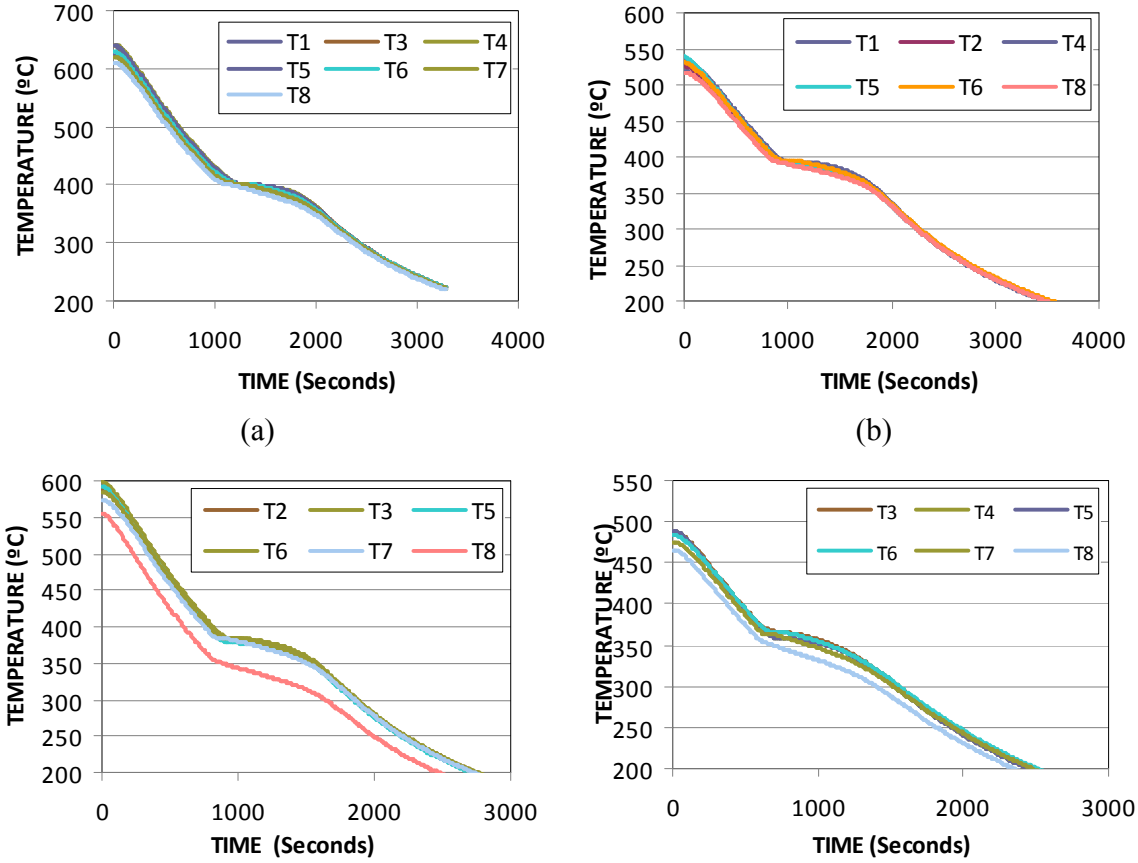
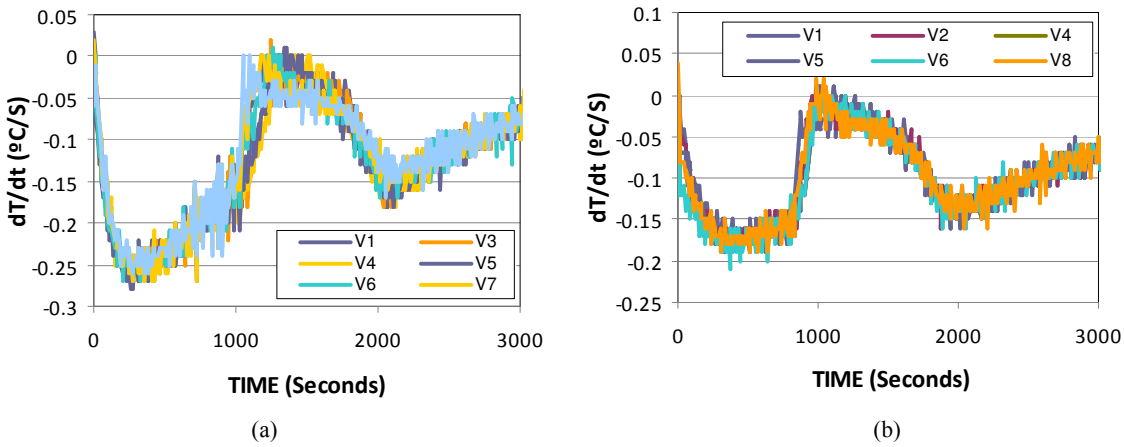
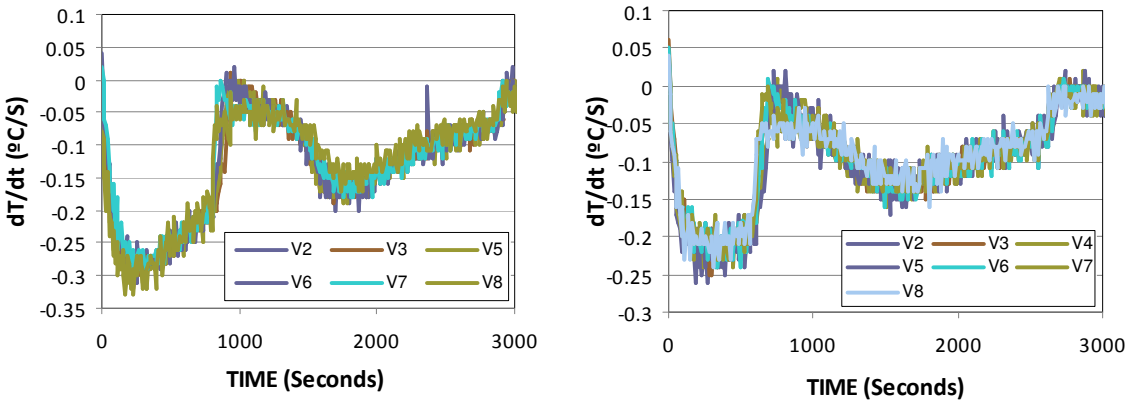


Figure 3. Temperature versus time curves.(a) Zn-4wt.%Sn, (b) Zn-6wt.%Sn, (c) Zn-10wt.%Sn, (d) Zn-30wt.%Sn alloys.

The cooling rate of the liquid alloy was determined from the temperature versus time curves at each thermocouple position (as can be observed in Figure 4 (a) to (d)) and taking the average slope.

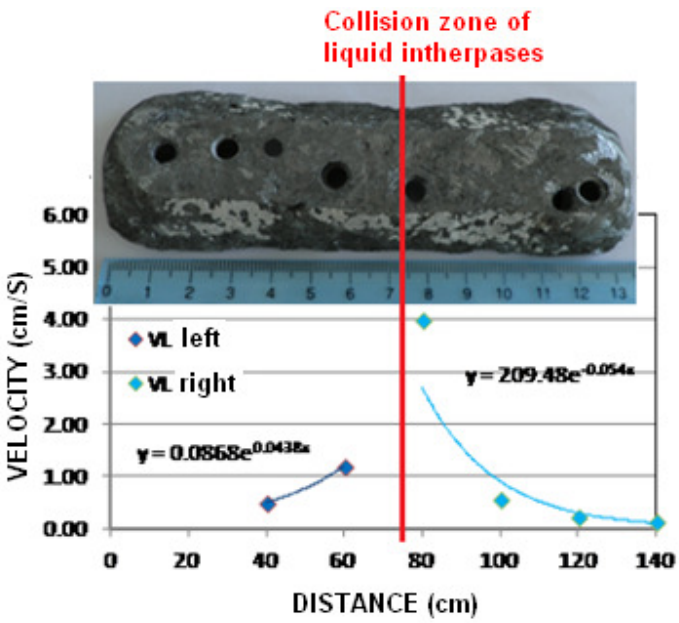




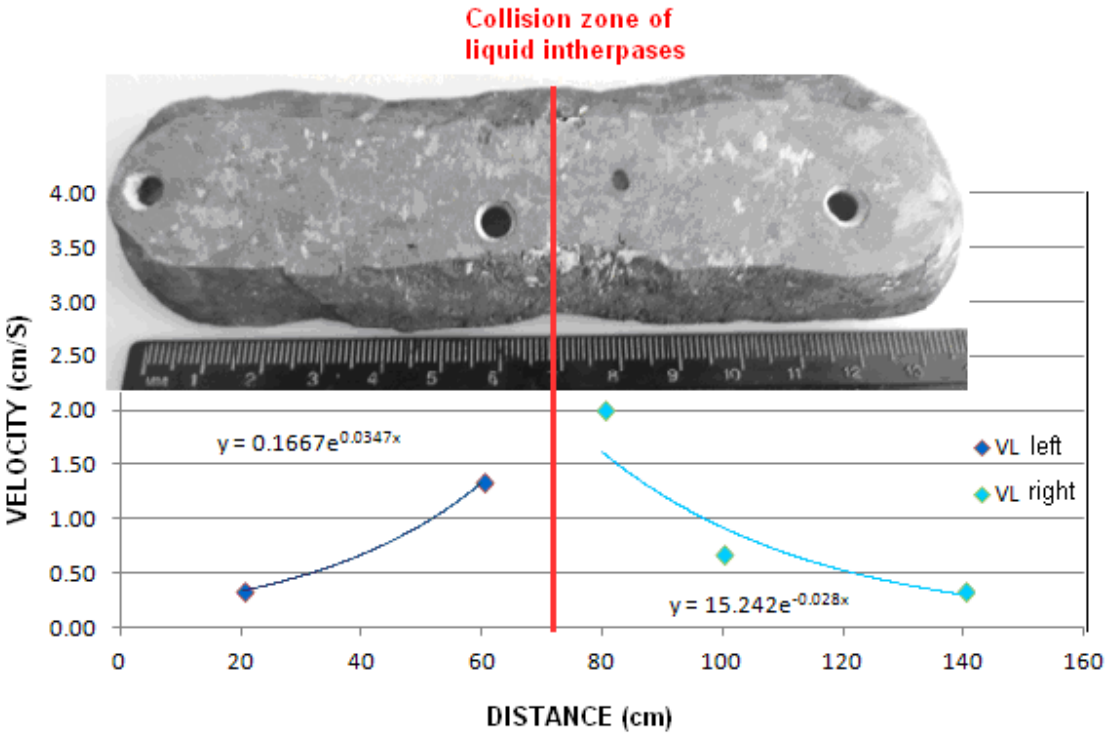
(c) (d)

Figure 4. Cooling rate versus time. (a) Zn-4wt.%Sn, (b) Zn-6wt.%Sn, (c) Zn-10wt.%Sn, (d) Zn-30wt.%Sn alloys.

The solidification velocities were determined by recording the start and the end of the of the liquidus and solidus temperatures at each thermocouple position; with these data it was possible to represent the position of the solidification front from T_L to T_S at each thermocouple position. From these types of figures the velocities and accelerations of liquid and solid interphases can easily be calculated, see Figure 5. In this case it can be observed an acceleration of the interphase movement.



(a)



(b)

Figure 5. Interphase velocity versus distance. (a) Zn-4wt.%Sn, (b) Zn-6wt.%Sn.

The values of temperature gradients are plotted in Figure 6 from both ends of the samples were the CET occurs. The minimum value always corresponds to the position of the change of the structure and that is the position of the columnar to equiaxed transition, CET.

The values of the critical gradients for each composition were (in Figure 6 are indicated with a red and rose-colored respectively): 0.01 °C/cm (right side) and 0.23 °C/cm (left side) for Zn-4wt%Sn, 0.25 °C/cm (right side) and 0.41 °C/cm (left side) for Zn-6wt%Sn, 1.4 °C/cm (right side) and 1.7 °C/cm (left side) for Zn-10wt%Sn and -0.05 °C/cm (right side) and 0.15 °C/cm (left side) for Zn-30wt%Sn.

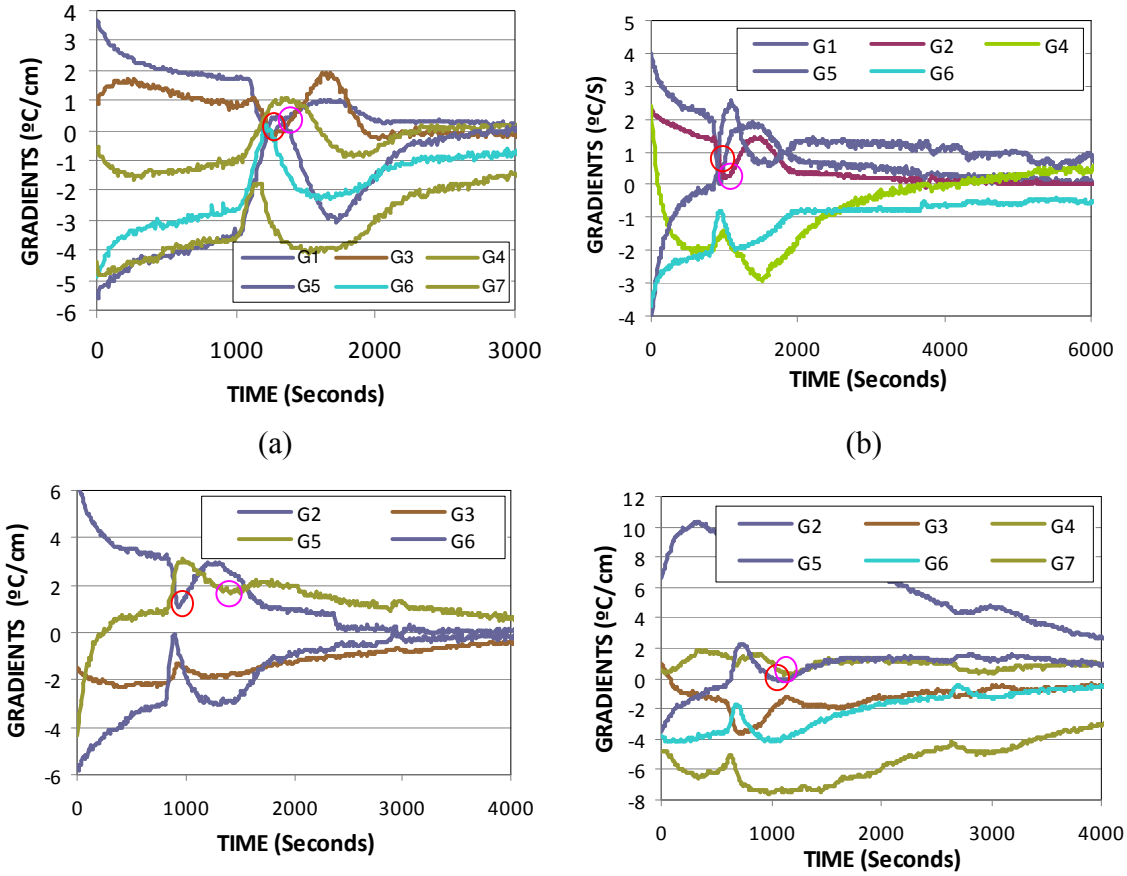


Figure 6. Temperature gradient versus time. (a) Zn-4wt.%Sn, (b) Zn-6wt.%Sn, (c) Zn-10wt.%Sn, (d) Zn-30wt.%Sn alloys.

Summary and Conclusions

As was previously reported for Zn-Sn diluted alloys (Zn-1wt%Sn to Zn-4wt%Sn) in a previous work the columnar-to-equiaxed transition (CET) was produced in a horizontal setup.

In the present work was determining the main parameters involved in the CET, in the case of Zn-4wt.%Sn, Zn-6wt.%Sn, Zn-10wt.%Sn and Zn-30wt.%Sn alloys, these parameters are:

- the moment of start and end of solidification at each position under consideration,
- cooling rates,
- the average temperature gradients and critical temperature gradients at the CET,
- the average speed of liquid and solid interphases left and right, advancing in opposite directions.

Also, solidification kinetics was found to be different between horizontal solidification two-way heat extractions, with preliminary results of the vertical unidirectional solidification.

In the solidification experiments, the velocity and accelerations of the liquidus and solidus interphases usually occurs at the end solidification zone of the samples.

Acknowledgements

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