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Research Paper

INFLUENCE OF PROCESS PARAMETERS ON MACROSEGREGATION OF DIRECTIONALLY SOLIDIFIED LEAD - TIN ALLOYS

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Directional solidification experiments were carried out on hypoeutectic and hypereutectic Pb-Sn alloys. Different combinations of growth rate V and composition Co. were used to investigate their effect on longitudinal macro segregation. Macro segregation along the length of the samples was observed in hypoeutectic Pb-Sn alloys whereas no such macro segregation was observed in hypereutectic alloys. The intensity of longitudinal macro segregation was found to increase with the increase in initial tin content of the alloy, increase in distance from the chill end and decrease in the solidification rate.

Keywords: Directional solidification, Macro segregation, Growth rate

INTRODUCTION

Macro segregation, the non-uniform macroscopic distribution of the components of an alloy during solidification, is a defect, which can occur in real metal processing systems. These non uniformities, especially when they form high compositional gradients, can be areas of high stress concentration, which cause cracking when an aluminum or steel billet is extruded or forged or when an as-cast piece, such as turbine blade, is severely loaded in service (Krane and Incropera, 1997).

The physics of macro segregation formation can be summarized as follows. Segregation starts at the microscopic level as solidification proceeds. During solidification, solute is rejected or depleted continuously from the precipitated solid and the composition of the surrounding liquid is consequently affected. If significant concentration gradients are developed at the interface, the interdendritic liquid can be driven simultaneously by thermal and solutal buoyancy, as well as solidification contraction. The induced flow will wash away the liquid next to the interface, resulting in

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segregation at the macroscopic level called macro segregation (Stefanescu, 2002).

When a homogeneous, stagnant fluid is subjected to a vertical temperature gradient, the resulting instabilities can give rise to convective flow patterns. However, if the resulting temperature at a particular location falls below the local liquidus temperature it is likely to promote solidification. In case of a multicomponent system a mushy layer of dendritic crystals may form during solidification, the interstices of which accommodate the residual solute that is rejected during the process. Thus in addition to the temperature gradient created by externally imposed boundary conditions as well as due to the release of the latent heat, concentration gradients are also created by the rejection of the solute across the solid/liquid interface. The rejected solute may be transported by diffusion on a local scale leading to micro segregation and by convective flow on a larger scale leading to macro segregation. From the practical viewpoint, the nature and extent of the macro segregation will determine the quality of the final product and hence forms an important research area.

If the melt is cooled from below, the fluid layer is always thermally stable, and hence, thermal buoyancy driven flow does not occur. Additionally if the system rejects a denser residual on solidification, both thermal and compositional fields are gravitationally stable, and convective motions do not occur (Huppert, 1990). Numerous theoretical, experimental and numerical studies have been devoted to the fluid mechanics aspects of solidification, which is presented as a comprehensive review by Huppert (Kumar *et al.*, 2003).

In a vertically upward growth, the fluid flow will be controlled by the density gradient in the melt at the interface. If this density gradient is positive, i.e., the rejected solute is lighter; the fluid flow effect can be significant. In contrast, when the axial density gradient is negative, i.e., heavier solute is rejected; fluid flow can still occur if any horizontal thermal gradient is present. Trivedi *et al.* (2001) have carried out a detailed numerical analysis of the effect of radial temperature gradient on the intensity of convection. In this research an attempt is made to examine the longitudinal macro segregation during upward directional solidification under controlled experimental conditions. The effect of solidification parameters on macro segregation is investigated.

EXPERIMENTAL PROCEDURE

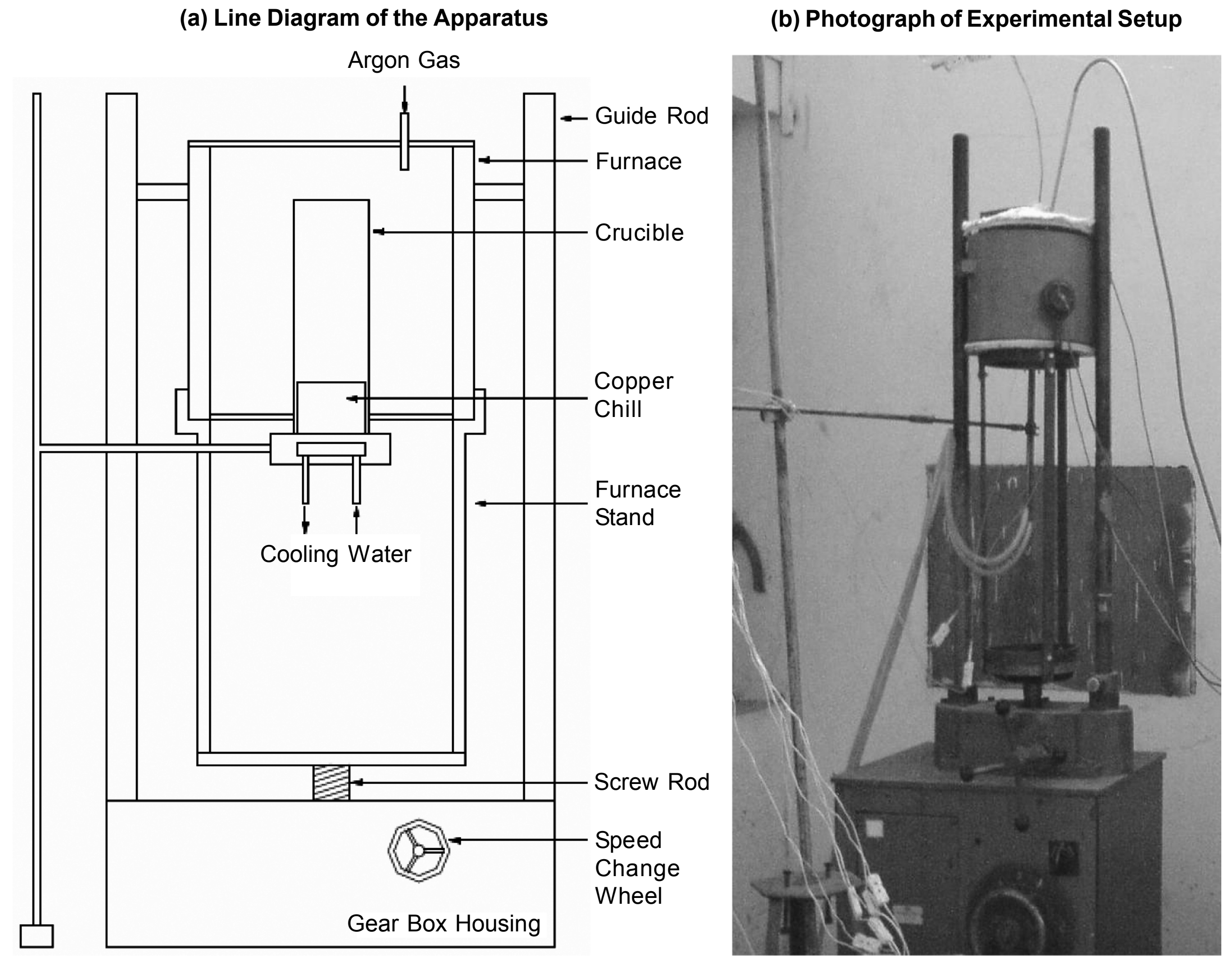
Alloy Preparation

Six different alloy compositions of Pb-Sn, three each in hypoeutectic (Pb-30Sn, Pb-40Sn, Pb-50Sn) and hyper eutectic (Pb-70Sn, Pb-80Sn, Pb-90Sn) ranges were prepared by remelting 99.9% Pb and 99.9% Sn using a high frequency induction furnace. Fully stirred melt was rapidly poured in to the cast iron mold and the alloy samples were obtained from this ingot.

Directional Solidification

The apparatus used for upward directional solidification is schematically illustrated in Figure 1a. Figure 1b shows the experimental setup. In an ideal directional solidification system, the imposed temperature gradient is perfectly perpendicular to the plane of

Figure 1: Directional Solidification Facility



solidification, which is the melt/crystal interface. Lateral temperature gradients are not desirable because they cause thermal stresses, which lead to the formation of vacancy, and dislocation defects in the crystals (Reza, 1994). The furnace temperature is designed to increase in the direction opposite to gravity in order to suppress natural convection. The rise in temperature opposite to gravity causes the density to decrease with increasing height.

The prepared alloy samples of 10 mm diameter and 50 mm long were placed in SiO_2 crucibles and melted. The molten samples were solidified directionally upwards in the experimental setup consisting of a heating unit, a temperature control system, a sample moving system and a heat extraction system. Initially the melt is allowed to reach the selected temperature and allowed to solidify from the bottom. The water-cooled copper chill inserted at the bottom of the SiO_2 crucible produced temperature gradient in the melt. The furnace can be moved upwards or downwards at a speed of 4-100 ìm/s by means of a translation device consisting of a set of gears and a motor. The directional solidification was carried out by raising the furnace assembly at various translation speeds with respect to the stationary sample. This avoids convection due to crucible motion. The furnace and the crucible arrangements were such that the furnace translation speed was equal to the directional solidification rate.

RESULTS AND DISCUSSION

The directionally solidified samples were longitudinally sectioned and polished for microscopic observation and examined using Scanning Electron Microscope. The SEM EDX spectrum showed the presence of macro segregation in hypoeutectic Pb-Sn alloys and the absence of any such segregation in hyper eutectic Pb-Sn alloys. Segregation results for Pb-Sn binary alloys are shown in Figures 2, 3 and 4 in which Cs/Co is plotted against distance from the chill end. Cs is the composition at the measured location of the sample and Co is the initial (average) composition of the sample.

Figure 2: Macro Segregation of Hypoeutectic Pb-Sn alloy Directionally Solidified at 100 μm/s

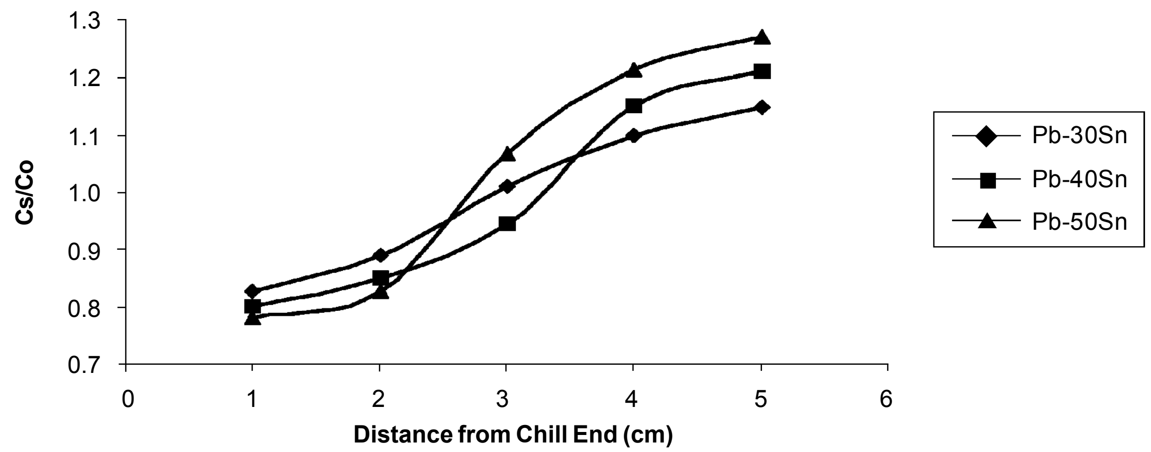


Figure 3: Macro Segregation of Hypereutectic Pb-Sn Alloy Directionally Solidified at 100 μm/s

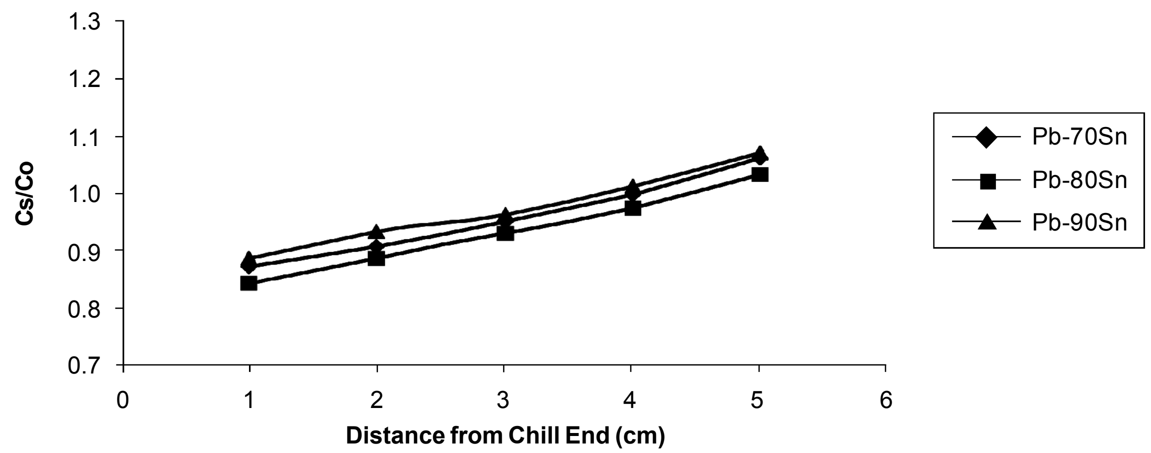
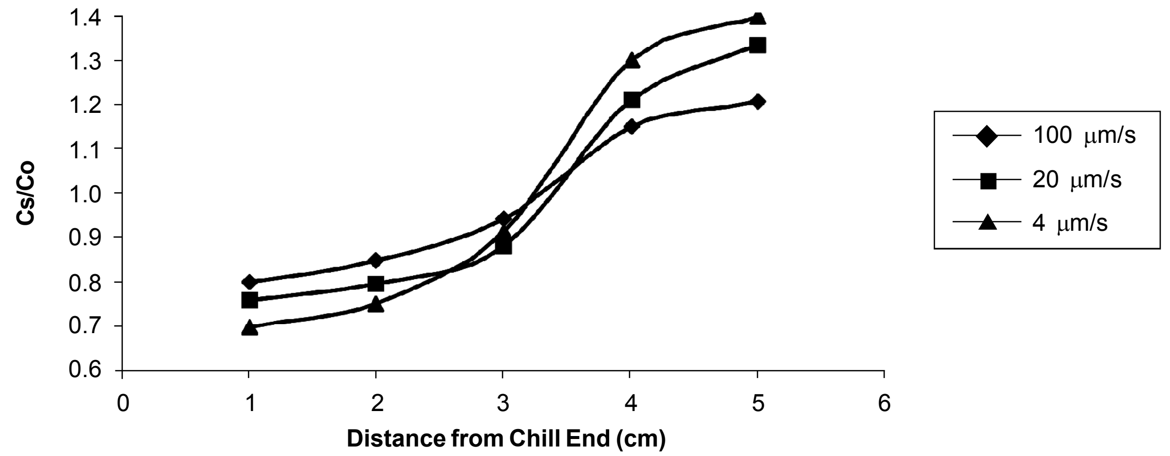


Figure 4: Effect of Growth Rate on Macro Segregation of Directionally Solidified Pb-40Sn Alloy



Figures 2 shows the macro segregation results of hypoeutectic Pb-Sn alloys. It shows that Cs/Co value increases with the increase in distance from the chill end, indicating the presence of macro segregation. The solidification begins at the bottom and progresses upwards. The rejected solute (Sn) at the solidification interface near the bottom gets redistributed in the remaining melt consequently increasing the tin content in the melt. Therefore the alloy that solidifies at the later stages will be of higher composition. Thus, as the solidification progresses upwards the solute content in the melt progressively increases giving rise to macro segregation in the longitudinal direction. It is also observed that the variation in the slopes of the three curves indicates that the macro segregation increases as the initial tin content in the alloy increases.

Figures 3 shows the macro segregation results of hypereutectic Pb-Sn alloys. It shows that the Cs/Co value is nearly the same along the entire length of the sample. This indicates the absence of any significant longitudinal macro segregation in case of hypereutectic Pb-Sn alloys for all the three values of Co considered. In the case of hypereutectic Pb-Sn alloys the primary dendrites are of tin and the rejected solute is lead. The density of lead is greater than that of tin, so the solute convection effects are minimized. Therefore the rejected solute atoms will not be carried away from the solid liquid interface, which minimizes the macro segregation.

Figure 4 shows the dependence of macro segregation on solidification rate. The intensity of macro segregation increase as the solidification rate decreases. The longitudinal segregation was greatest in the slowest velocity samples. At the slowest velocity of 4 μm/s, there is sufficient time for transport of species by both diffusion and advection in the liquid.

Macro segregation is associated with the effect of gravity. The convection driven by vertical solutal gradient leads to macro segregation. In the case of hypoeutectic Pb-Sn alloys the solute (Sn) is lighter than the solvent (Pb). As the crystals of the primary dendrite (Pb) form, the solute (Sn) in the melt surrounding the primary dendrite increases and the density profile in the interdendritic melt and in the melt immediately ahead of the mushy zone promotes natural convection. In the case of hypereutectic Pb-Sn alloys the solute (Pb) is heavier than the solvent (Sn). As the crystals of primary dendrite (Sn) form, heavier solute (Pb) in the melt surrounding the primary dendrite and in the melt immediately ahead of the mushy zone prevents natural convection. In this case both the solutal gradient and the thermal gradient stabilize the liquid system. In upward directional solidification thermal gradient opposes the solutal gradient. The thermal Rayleigh number (RT) and solutal Rayleigh number (RC) have been defined in the literature to describe the onset of the thermosolutal convection in the melt (Heinrich *et al.*, 1989). The two numbers are given as:

$$R_T = g\alpha G_T H^4/K_T \nu$$

$$R_C = g\beta G_C H^4/D_C \nu$$

Where α is the thermal volume expansion coefficient, g the gravitational acceleration constant, G_T the thermal gradient, K_T the thermal diffusivity, ν the kinematic viscosity, β the solutal volume expansion coefficient, G_C the solutal gradient, D_C the solutal diffusivity and H a characteristic length (for the dendritic arrays, the primary arm spacing has been used as the characteristic length). Data listed in Table 1 (Sazarin and Hallawell, 1988) were used to calculate the R_T and R_C under the conditions of this research. $R_T = 4.2 \times 10^8 (G_T) H^4$, $R_C = 6.8 \times 10^{13} (G_C) H^4$, $R_C/R_T = 1.61 \times 10^5 (G_C/G_T)$. The solutal Rayleigh number determines the intensity of macro segregation when the thermal gradient (G_T) is not excessively large, because the solutal Rayleigh number is much larger than the thermal Rayleigh number.

Table 1: Data Used in the Calculation of Rayleigh Number for Pb-Sn Alloy

Properties	Value
Kinematic viscosity of melt (ν)	2.47×10^{-7} (m^2s^{-1})
Thermal coefficient of volumetric expansion of melt (α)	1.15×10^{-4} (K^{-1})
Solutal coefficient of volumetric expansion of melt (β)	5.20×10^{-3} (wt%$^{-1}$)
Thermal diffusivity of melt (K_T)	1.08×10^{-5} (m^2s^{-1})
Solutal diffusivity of melt (G_C)	3.00×10^{-9} (m^2s^{-1})
Acceleration due to gravity (g)	9.80×10 (ms^{-2})

Figures 5a and 5b show the optical micrographs of the hypoeutectic and

Figure 5: Microstructures Alloy Samples Directionally Solidified at 100 μm/s

(a) Hypoeutectic (Pb-50Sn)

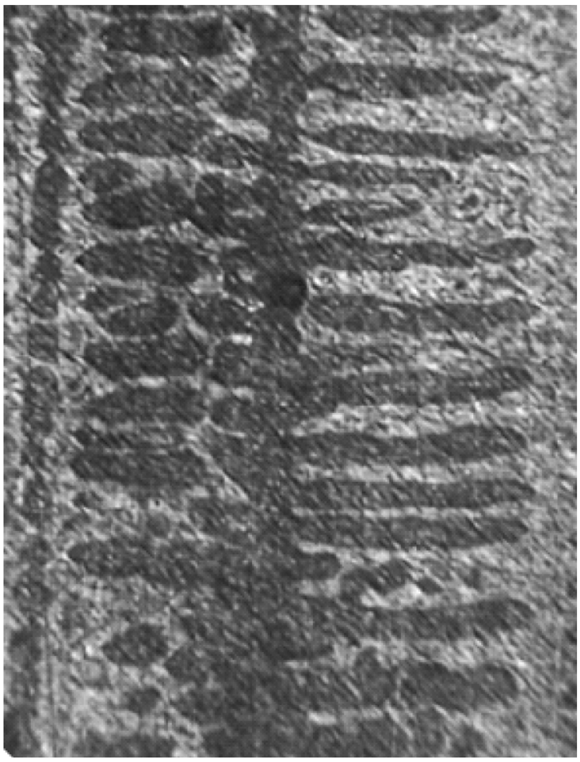
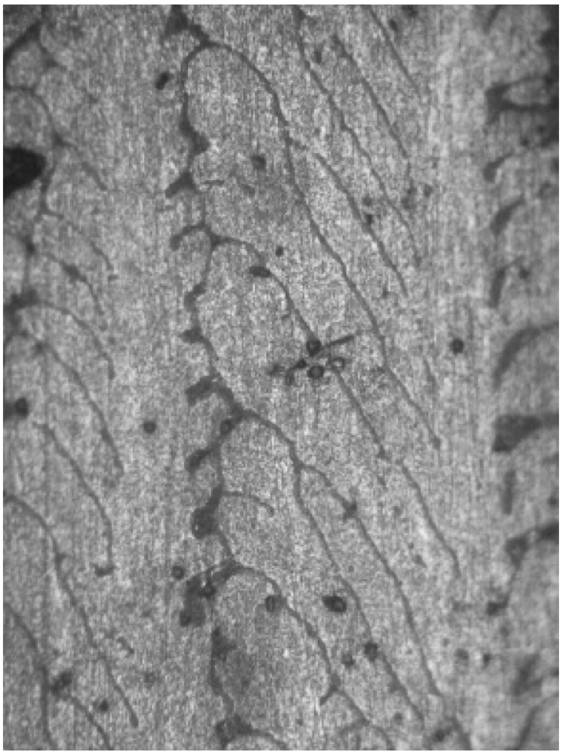


Figure 5 (Cont.)

(b) Hypereutectic (Sn-10Pb)



hypereutectic Pb-Sn alloys respectively. The figures clearly show that, in case of hypoeutectic Pb-Sn alloys the primary dendrite is of lead whereas in case of hypereutectic Pb-Sn alloys the primary dendrite is of tin, which is confirmed by SEM EDX.

CONCLUSION

Upward directional solidification experiments were carried out using hypoeutectic and hypereutectic Pb-Sn binary alloys with different combinations of growth rate and composition. The effect of solute convection on the macro segregation was investigated. The following conclusions were drawn from the study

- Convection during upward directional solidification produces extensive longitudinal macro segregation.
- When the solute is lighter than the solvent, macro segregation results from the convection driven by the vertical density gradient.
- Macro segregation increases as the solute content in the alloy increases.
- The intensity of macro segregation increases as the solidification rate decreases.
- When the solute is heavier than the solvent, macro segregation is not observed due to the absence of convection.

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