Effect of melt heat treatment on the solid/liquid interface morphology of directional solidification

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Received 14 June 1999

Abstract

A set of quadril-electrode DC input voltage measure equipment was built up to detect the electrical resistance of Sb–Bi single-phase alloy. The results suggest that Sb–5 wt% Bi undergoes structural transition in the temperature range of 730–750°C. By overheating the melt to different temperatures and then cooling to 690°C after a period of time, the samples are directionally solidified in a Bridgeman-type furnace. It is found for the first time that after heat treatment of the melt, the solid/liquid interface morphology shows significant variation depending on the specific heat treatment. Also, in Ag–Cu, Al–Cu systems, bicrystal growth with different orientation is found to have different interface morphologies after heat treatment. It is believed that the evolution of the cluster size distribution in the melt contributes to these phenomena.

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Keywords: Melt heat treatment; Interface morphology; Cluster size distribution

1. Introduction

Heat treatment is used widely in solid transformation to improve material properties. It is also well known that heat treatment can eliminate heterogeneous nuclei, thereafter refining primary phase morphology and reducing casting porosity [1–7]. Melt heat treatment can be summarized as follows: Following the correlation between melt structure and temperature and the way of evolution during heating and cooling, melt heat treatment is the process to improve casting microstructure and performance by employing a heat effect to artificially control the melt structure and its variation.

In Russia, so-called BTO processing has been commonly applied as practical examples of melt heat treatment, which greatly improves the properties of superalloy and saves production costs [1–4]. Chinese scientists and others abroad have done many investigations in Al–Si, Al–Cu alloys, etc. and found great benefits [6,7]. However, similar research has seldom, if ever, been done in directional solidification. The effect of temperature on directional solidification is only regarded according to temperature gradient and cooling rate. In this paper, we will explore the effects of melt heat treatment on directional solidification.

2. Experimental procedure

Single-phase alloys Sb–5 wt% Bi and Ag–1% wt Cu were selected for this research. No primary phase will form during the solidification of these alloys, which simplifies the solidification process. Samples are cast in a vacuum induction furnace with argon atmosphere.

To detect melt structure variation, a set of quadril-electrode equipment was developed, as shown in Fig. 1. So far, only the electrical resistance of Sb–5% wt Bi alloy has been measured. A standard DC current of 10 mA is introduced into the melt, the voltage output being measured by a pen recorder with a sensitivity of 0.5 μV/cm. The temperature of the melt is detected by a pair of thermocouples. To prevent alloy evaporation, the whole crucible is enclosed by iron seals. Experimental results during heating and cooling are shown in Fig. 2.

It can be seen that the value of resistance undergoes a transition regime in the temperature range from 730 to 750°C during heating. This is consistent with DTA analysis results, which show an exothermic peak at 731.58°C. Thus, during directional solidification, the melt heat treatment temperatures were chosen as 690, 735 and 1000°C. Each sample was kept at its corresponding temperature for 30 min, then cooled to 690°C in 10 min, and finally, withdrawn at 1.5 μm/s in a Bridgeman-type furnace. The microstructures, thus obtained, are shown in Fig. 3.

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PII: S1468-6996(01)00043-2
Fig. 1. Illustration of the electrical resistance measurement equipment.

![Illustration of electrical resistance measurement equipment](image1)

Fig. 2. Electric resistance versus temperature of Sb–5wt% Bi melt.

![Graph of electric resistance vs. temperature](image2)

Fig. 3. Microstructures of Sb–5wt% Bi withdrawn at 1.5 μm/s after melt heat treatment at: (a) 690°C, (b) 735°C, and (c) 1000°C for 30 min, then cooled down to 690°C in 10 min.

![Microstructures of Sb–5wt% Bi](image3)
For Ag–1wt% Cu, electrical measurements have not yet been done, so the melt heat treatment temperatures were chosen somewhat arbitrarily. Overheat of the melt above its liquidus temperature were chosen as 300, 500 and 700°C. No cooling down to a lower melt temperature was made, rather the samples were directly withdrawn at the overheated melt temperature. Some results are shown in Fig. 4. From the comparison of the original microstructures with those of quenched microstructures, it is found that the original interface is not preferred oriented. By X-ray diffraction, it is found that the higher the temperature of the melt, the more complicated are the interface orientations, as listed in Table 1.

3. Discussion

After melt heat treatment, all Sb–Bi samples are solidified under the same temperature gradient and traction rate. According to traditional solidification theory, they must have the same microstructure. However, it is seen in Fig. 3 that melt heat treatment changes the interface morphology significantly. At 690°C, the sample without heat treatment is not even directionally solidified. The sample heated to 735°C shows a cellular microstructure with facetted tips, while that heated to 1000°C exhibits dendritic morphology. This cannot be explained by heat treatment theory of heterogeneous nuclei being dissolved by heating. Thus, no theory

| Melt temperature (°C) | Interface velocity (μm/s) | Interface orientation | Inclination angle with the polished face |
|-----------------------|---------------------------|-----------------------|----------------------------------------|
| 1100                  | 1                         | [111]                 | –                                      |
| 1167                  | 1.7                       | [110]                 | –                                      |
| 1230                  | 2                         | [111]                 | 8.6                                    |
| 1260                  | 1                         | [110]                 | 14                                     |
| 1435                  | 4                         | [111]                 | –                                      |
| 5                     | [111][100]                | 4.4                   |
| 10                    | [111]                     | –                     |
| 1460                  | 3                         | [111]                 | 0                                      |
| 1610                  | 1                         | [110]                 | –                                      |
| 3                     | α: [111]                  | α = 14.8              |
| β: [200][220][311]    | β = 4.6                   |
| 6                     | α: [111]                  | –                     |
| β: [200][220][311]    |                           |
so far developed can explain this phenomenon successfully. The authors can only suggest that even simple metallic melts, such as pure metals or solutions, are composed of clusters rather than single atoms [8]. The clusters have some size distribution (CSD), which depends on the temperature. CSD will affect the interface attachment process and furthermore the interface morphology.

From that observed in Ag–1wt% Cu (Fig. 4), it is clear that at high melt temperature, those orientations with high index, which cannot grow at low melt temperature, can grow stably. Two crystals with different orientation also exhibit different morphology. The latter may be ascribed to interface anisotropy, but the former must be due to CSD.

Detailed explanation of melt heat treatment effects awaits further study of liquid structures.

4. Conclusion

The concept of melt heat treatment is advanced. In Sb–5wt% Bi alloy, a set of quadral-electrode equipment was built up to detect electrical resistance transition of the melt. The results suggest that structural transition occurs at around 730°C. A kind of melt heat treatment is applied to Sb–Bi and shows significant effect on interface morphology in directional solidification for the first time, while in Ag–1wt% Cu, the samples are solidified under different melt temperatures, which prove that high index orientation can grow stably at high melt temperature. Traditional theory cannot explain these phenomena. The authors believe that CSD in the melt is related to these observations.

References

[1] S.M. Voronov, Selected Papers on Light Alloys, Moscow, 1957, p. 357.
[2] V.I. Nikitin, et al., The sedimentation of liquid aluminium alloys depending on the charge composition, Soviet Casting Production 4 (1991) 12–13.
[3] V.I. Nikitin, P.S. Popel, A.M. Paramonov, et al., Modifying aluminium alloys with consideration for the structural heredity phenomenon, Soviet Nonferrous Metals 9 (1992) 63–66.
[4] V.I. Nikitin, 60th World Foundry Congress, vol. 35, 1993, p. 1.
[5] G.H. Vineyard, Liquid Metals and Solidification, ASM, Cleveland, 1958, p. 1.
[6] P. Li, M. Gui, J. Jia, et al., Electrical resistance of Al–16wt% Si and its structural transition, Zhu Zao 9 (1995) 15–20 (in Chinese).
[7] L. Zhang, X. Bian, J. Ma, Liquid structural transition of Al–Si alloy, Zhu Zao 10 (1995) 7–12 (in Chinese).
[8] Y. Cai, X. Fan, J. Li, H. Fu, On the effect of clusters in crystal growth from the melt, Acta, Northwestern Polytechnical University 14 (1996) 323–324 (Xi Bei Gong Ye DaXue Xue Bao).