Microstructure evolution and coarsening kinetics of semisolid CuSn10P1 alloy under short-time isothermal treatment

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Keywords: CuSn10P1 alloy, semisolid, short-time isothermal, microstructure evolution, coarsening kinetics

Abstract

The key success of semisolid rheology is to prepare high-quality semisolid slurries. To achieve that it is necessary to be able to characterize the microstructure, element distribution, and coarsening mechanism during short-time isothermal treatment. This present work applied the enclosed cooling slope channel (ECSC) to make a semisolid slurry of CuSn10P1 alloy. Effects of short-time isothermal treatment (0, 5, 10, 15, 20, 25 s) on microstructure characteristics, element distribution, grain stability, and coarsening kinetics of semisolid CuSn10P1 alloy were studied. The equiaxed or near-globular microstructure can be prepared and improved intergranular segregation of CuSn10P1 alloy through the semisolid slurry short-time isothermal treatment. The mean diameter of primary α-Cu phase gradually increases with soaking time prolonged, but shape factor shows a wavy trend. The mean size and shape factor changes caused by the instability of primary α-Cu phase during short-time isothermal treatment can be described by the melting mechanism and the coarsening mechanism. The relationship between the high Tin layer thickness of primary α-Cu and soaking time is consistent with the linear equation. The relationships of the Sn mass fraction in intergranular microstructure and primary α-Cu phase center versus soaking time are consistent with an exponential equation. The suitable isothermal treatment parameters for the semisolid CuSn10P1 alloy is the soaking time 15 s to 20 s at 990 °C.

1. Introduction

The use of tin bronze has a long history and shows different properties with the change of tin element content [1]. Generally speaking, Tin bronze with lower tin element content (≤5%) has good conductivity, so it is widely used in wires and electronic devices [2]. With the increase of the content of Sn element (5%–9%), the tin bronze alloy is suitable for making springs, relay sockets, and mouse shrapnel because of its electrical conductivity and higher elastic modulus [3]. Tin bronze has the characteristics of high strength, large elastic modulus, good wear resistance, and corrosion resistance, as well as good fluidity when the content of Sn element is greater than 10%, which is widely used in castings of the marine and mechanical industries [4]. The microstructure of conventionally cast Tin bronze castings is dominated by coarse dendrites [5], which leads to a decline of casting properties due to defects such as shrinkage porosity, segregation, and other defects during the solidification process, it cannot meet the requirements of increasingly harsh working conditions [6, 7].

The semisolid processing (SSP) has been widely studied because its formed parts have the advantages of uniform microstructure, fewer defects, high properties, etc [8]. The process has been widely used in industrial production with the development of SSP. The semisolid parts have the advantages of good dimensional
precision, compactness, and near-net-shaped compared with traditional casting methods [9]. Semisolid forming process includes thixoforming and rheological forming. The transportation of the semisolid billets prepared by the thixoforming method is convenient, but the preparation process is complicated and increases the production cost [10]. Besides, solid-liquid separation is easy to occur during the forming process, making the uneven microstructure distribution in different positions of the part [11]. Rheological forming has become a research hotspot in recent years due to its high efficiency and low cost [12]. The rheological forming of aluminum [13, 14] and magnesium [15] has been extensively studied and promoted in industrial production. At present, there are some reports on the rheological forming of high melting point alloys such as steel [16] and copper [17], but the main research is the effect of semisolid slurry process parameters on slurry quality and casting properties. Short-time isothermal treatment can improve the microstructure uniformity of semisolid slurry [12]. For tin bronze alloys that are prone to intergranular segregation, the effect of short-time isothermal treatment on the microstructure uniformity and intergranular segregation of high melting point tin bronze alloys is still unclear.

In order to obtain high-quality semisolid slurry suitable for rheological forming, this paper adopts the self-developed enclosed cooling slope channel (ECSC) to make a semisolid slurry of CuSn10P1 alloy [18]. Effects of short-time isothermal treatment (0, 5, 10, 15, 20, 25 s) on microstructure characteristics, element distribution, grain stability, and coarsening kinetics of semisolid CuSn10P1 alloy were investigated.

2. Experimental method

2.1. Material
The composition of CuSn10P1 alloy is shown in table 1. Figure 1(a) shows the DSC curve of CuSn10P1 alloy. The solidus and liquidus temperatures of the alloy were 839.3 °C and 1024.3 °C, respectively. The curve between solid fraction and temperature was obtained according to the DSC curve calculation [19, 20], as shown in figure 1(b).

| Table 1. Composition of CuSn10P1 alloy (wt%). |
| Cu  | Sn  | P  | other |
|-----|-----|----|-------|
| 89.15 | 9.97 | 0.71 | 0.17 |

2.2. Short-time isothermal treatment process
The semisolid slurry was prepared by ECSC equipment [18] then isothermal treatment at 990 °C, the schematic of the ECSC equipment as shown in figure 2. It is suitable for the rheology forming of solid fractions is 24% at the isothermal temperature is 990 °C (figure 1(b)). The effects of soaking time (0 s, 5 s, 10 s, 15 s, 20 s, and 25 s) on microstructure characteristics, element distribution, grain stability, and coarsening kinetics of semisolid slurry were studied. The specimens were 10 × 5 × 5 mm³ bulks from semisolid slurry quenched immediately after reaching the soaking time.
2.3. Microstructure characterization
The specimens under different soaking times were with ground 200, 400, 600, and 800 grit sandpapers, use 0.5 μm diamond paste for mechanical grinding, and then use 5% FeCl₃ to etched for 3 ∼ 5 s. The microstructure of semisolid specimens under different soaking time was observed by an optical microscope (OM) and scanning electron microscope (SEM). The element distribution and phase identification of semisolid specimens was analyzed with quantitative energy dispersive x-ray spectrometer (EDS).

In this work, the average diameter (D) and shape factor (F) was statistical and analyzed by Image-J software according to the following equations [21, 22]

\[ D = \frac{\sum_{i=1}^{n} \frac{2A_i}{P_i}}{n} \]  
\[ F = \frac{\sum_{i=1}^{n} \frac{4\pi A_i}{P_i^2}}{n} \]

Where A is grain area; P is grain perimeter; n is the number of grains.

3. Results and discussion
3.1. Microstructure evolution during short-time isothermal treatment
Figure 3 shows the semisolid microstructure evolution of CuSn10P1 slurry with soaking time prolonged from 0 s to 25 s. The microstructure includes primary α-Cu phase and intergranular microstructure (α + δ + Cu₃P) phase. The mean grain diameter of primary α-Cu phase increases and distributes uniformly with prolonged soaking time.

The morphology is mainly equiaxed or fine dendrites without short-time isothermal treatment. However, the microstructure shows obvious agglomeration (figure 3(a)). The temperature field distribution of semisolid slurry in the short-time isothermal treatment crucible is unevenly just after prepared [23]. The temperature where the primary α-Cu phase agglomeration is lower, while liquid agglomeration is higher. The primary α-Cu phase is freed to achieve the purpose of uniform temperature field, and concentration field, and microstructure distribution. There is still obvious primary α-Cu phase agglomeration and fine dendritic microstructure formed by the liquid due to the short soaking time and primary α-Cu phase is not insufficiently freed (figure 3(a)).

The temperature field and concentration field will be more uniform by driven the temperature gradient and concentration gradient when the semisolid slurry isothermal treatment 5 s. The primary α-Cu phase will continue to dissociate to make its distribution more uniform than without isothermal treatment. The relatively uniformly distributed primary α-Cu phase is surrounded by liquid, and nucleation in liquid forms fine dendrites distributed around primary α-Cu phase during solidification, as shown in figure 3(b). The temperature field and composition field are more uniform with the isothermal treatment time increases 10 s, the primary α-Cu phase is evenly distributed in melt, and homogenization of the temperature field and composition.
field also promotes formation of new α-Cu phase grains in the melt. The solidified microstructure includes equiaxed crystals of different sizes and fine dendrites (figure 3(c)).

The primary α-Cu phase enters the stage of growing up and coarsening after soaking time for 15 s. The fine grains begin to grow or merged, and a small number of fine dendrites appear to be remelted from the roots of the dendrite arms, results in a large vary in α-Cu phase size (figure 3(d)). The new grains formed in the liquid and the fine grains formed by the remelting of the dendrites arm begin to grow when soaking time for 20 s, and primary α-Cu phase morphology is nearly spherical or rose petal-like with relatively uniform size and distribution (figure 3(e)). The primary α-Cu phase has obvious Oswald ripening and coarsening causes coarse grains but uniform distribution when the soaking time for 25 s (figure 3(f)).

Figure 4 reveals variations of solid-grain boundaries for semisolid CuSn10P1 slurry with the soaking time of 5 s, 15 s, and 25 s at 990 °C, respectively. The yellow arrow indicates the area where the dendrite arm remelting occurs, the blue ellipse indicates the fine dendrite formed in the liquid, the red ellipse indicates the completion of the dendrite arm remelting, and the yellow dashed ellipse indicates the primary α-Cu phase merge and coarsening. Obviously, with the prolonged soaking time of semisolid slurry from 5 s to 25 s during the isothermal treatment, the temperature field and concentration field changed from confusion to uniform, and the growth mechanism of primary α-Cu phase changed from melting mechanism to aggregation and Oswald ripening mechanism [10].

The temperature and concentration field are not uniform resulting in element accumulation at the front of solid-liquid interface and dendrite root causes remelting of the dendrite root when soaking time for 5 s, the areas with high temperature and more liquid are easy to form fine dendrites when solidified (figure 4(a)) [24]. The temperature field and the concentration field become more uniform as the soaking time is prolonged to 15 s. At
this time, the melting mechanism and coarsening mechanism coexist. The dendrite root remelting will continue to occur where there is solute enrichment at the root of the dendrite, while the fine grains grow up and coarsen or merged (figure 4(b)). The temperature field and concentration field are uniform when the soaking time for 25 s, the grain growth is mainly based on aggregation and Oswald ripening (figure 4(c)). This process is consistent with the preparation of A356 alloy semisolid slurry by cooling slope processing [25] and Gas-induced semisolid (GISS) [12]. The remelting of dendrite arm is caused by a local temperature too high or solute enrichment and infiltration during the short-time isothermal treatment process, while the coarsening of the grains is driven by the reduction of interface energy because of the decline of interface area [10]. The refinement of primary α-Cu phase increases the interface energy and free energy of the system, and the system will regularly reduce the interface energy to ensure the stability of the system. Therefore, the large primary α-Cu phases merge with the small ones to become coarse and spheroidized under the drive of the interface energy. However, the relatively higher temperature of the isothermal treatment and the solid fraction is only 24%. The roundness of primary α-Cu phase variations continuously during short-time isothermal treatment because of the melting and the coarsening mechanism coexist.

3.2. Element distribution during isothermal treatment
In order to avoid the influence of grain size on the diffusion depth and content of the Tin element when studying the effect of isothermal treatment at 990 °C on the diffusion of Tin element, select similar equivalent diameters of primary α-Cu phase for research. Figure 5 shows the line scanning of CuSn10P1 semisolid slurry during the different soaking time at 990 °C. The microstructure includes white eutectoid (α + δ + Cu3P) phase and black primary α-Cu phase and gray-white high Tin layer. Line scan shows that the content of Tin in the intergranular microstructure is the highest, while primary α-Cu phase is the lowest. The diffusion of Cu and Tin elements is mainly affected by the diffusion coefficient, and the relationship between the diffusion coefficient and temperature follows the Arrhenius equation [26]:

\[ D = D_0 \exp \left( -\frac{Q}{RT} \right) \]  

Equation (3) shows the melt temperature directly determines the diffusion coefficient. The melt temperature is constant during the isothermal treatment, therefore, the diffusion rate of Cu and Tin elements is unchanged. The thickness of the transition layer increases from 1.37 μm at 0 s to 4.39 μm at 25 s as the soaking time increases at 990 °C isothermal treatment. Figure 6 shows the fitting curve of transition layer thickness (H) versus soaking time (t). The thickness of transition layer has a linear relationship with the soaking time during the isothermal treatment \( (R^2 = 0.998) \), as shown in equation 4.

\[ H = 0.12t + 0.73 \]  

Figure 7 shows the microstructure and SEM of conventional casting CuSn10P1 alloy. It can be seen from figure 7 that the microstructure of conventional casting is coarse and dendritic, and the phase composition is the same as that of the semisolid slurry, but there are a lot of shrinkage cavities and porosity defects.

The composition of points 1, 2, and 3 in figures 5 and 7 are shown in table 2. Point 1 is the composition of intergranular microstructure, and point 2 is the composition of small plateaus that appear in the transition layer, and point 3 is the composition in primary α-Cu phase. Tin element content of intergranular microstructure (point 1) continuous decline, but steadily increased in primary α-Cu phase (point 3) during the isothermal treatment. The mass fraction of the Tin element at point 2 fluctuates around 15.8 (wt%), which may be peritectic β\(^\prime\) phase [27]. The content in the center of primary α-Cu phase is 1.72% (wt%) increased to 3.59% (wt%) due to
Figure 5. SEM-EDS analysis of CuSn10P1 semisolid slurry during different soaking time at 990°C: (a) 0 s, (b) 5 s, (c) 10 s, (d) 15 s, (e) 20 s, (f) 25 s

Figure 6. Transition layer thickness of primary $\alpha$-Cu phase as a function of soaking time at 990°C.
Tin element diffuses during isothermal treatment. Simultaneously, the thickness of the transition layer with high Tin elements has also been significantly increased. The short-time isothermal treatment significantly improves the intergranular segregation phenomenon.

Figure 8 shows the relationship between the Tin element mass fraction in the intergranular microstructure and primary \(\alpha\)-Cu phase versus the soaking time. The Tin element content of intergranular microstructure (point 1) and primary \(\alpha\)-Cu phase (point 3) has an exponential relationship with the soaking time. The fitting formulas are shown in equations (5) and (6), respectively.

\[
Y_i = 23.315e^{-0.046t}
\]  
(5)

\[
Y_p = 0.0043t^{1.805} + 1.628
\]  
(6)

Tin bronze is easy to form intergranular segregation during conventional casting, but semisolid processing can significantly refine the primary \(\alpha\)-Cu phase and increase its surface area. Thereby increasing the diffusion area of Tin element and shortening the diffusion couple distance. Short-time isothermal treatment of semisolid slurry not only homogenizes the microstructure but also reduces the degree of intergranular segregation. Therefore, the short-time isothermal treatment of semisolid slurry can improve the uniformity of microstructure and casting properties.

3.3. Growth kinetics of primary \(\alpha\)-Cu phase

The primary phase has two competing mechanisms of the melting and coarsening during the semisolid microstructure evolution \cite{28, 29}. The melting mechanism is because of local high-temperature fusing or the infiltration of solute enrichment in the melt, which suppresses the growth of primary phase \cite{28}. Grain coarsening is mainly controlled by grain aggregation or Oswald ripening mechanism, thereby controlling the growth of solid-phase grains \cite{29}. The two competition mechanisms affect the growth of primary phase and further determine the mean size and shape factor of primary phase. Therefore, the strength of two competition mechanisms determines the mean size and shape factor of primary phase during the isothermal treatment of semisolid slurry \cite{30}.

Figure 7. Microstructure and SEM of conventional casting CuSn10P1 alloy.

Figure 8. Sn mass fraction in intergranular microstructure and at the center of the primary \(\alpha\)-Cu phase as a function of soaking time at 990 °C.
Langer proposed a model for the stable growth of grain interfaces based on the Mullins-Sekerka (M–S) instability theory [31, 32].

\[
R_c = \left( \frac{30D_k \Gamma}{m_L C_0 (k - 1) \nu} \right)^{\frac{1}{2}}
\]

(7)

Where \( R_c \) is the critical particle radius; \( C_0 \) is the bulk solute concentration; \( \nu \) is solid–liquid interface growth rate; \( \Gamma \) is Gibbs–Thompson modulus; \( D_k \) is the solute diffusion modulus in melt; \( m_L \) is liquidus slope; \( k \) is distribution constants. The solid–liquid interface growth rate is as follows.

\[
\nu = \frac{R_m}{t}
\]

(8)

The \( t = 5 \text{ s} \) in this study, the figure 9 shows the relationship curve of particle radius versus interface velocity obtained from the data in table 3 [33–36]. The calculated particle size is on the dividing line, indicates that the phenomenon of melting and coarsening of primary \( \alpha – \text{Cu} \) phase occurs at the same during the isothermal treatment process.

Figure 10 shows the average size and shape factor of the primary \( \alpha – \text{Cu} \) phase in the semisolid slurry at different soaking times. The mean grain size gradually increases from 45.1 \( \mu \text{m} \) to 59.8 \( \mu \text{m} \) when the CuSn10P1 semisolid slurry is soaking time from 0 s to 25 s. The mean size of primary \( \alpha – \text{Cu} \) phase represents increasing in short-time isothermal treatment, which corresponds to those research results of gas-induced semisolid (GISS) processed 356 aluminum alloy [12] and low superheat pouring with a shear field (LSPSF) formed AlSi9Cu3 semisolid slurry [37]. Furthermore, the mean size does not change between soaking 15 s and 20 s. The reason maybe is that the small grains at soaking 20 s are melted or coarsen which quickly consumes the Tin element in liquid and inhibits rapid growth of surrounding large grains.

The roundness represents the wavy trend with the soaking time increasing from 0 s to 25 s at 990 °C. This corresponds to the experimental result of GISS processed 356 aluminum alloy [12] and semisolid isothermal treatment of wrought superalloy (SSITWS) GH4037 alloy [10]. The temperature field and concentration field are first quickly homogenized during short-time isothermal treatment, and large \( \alpha – \text{Cu} \) grains are freed, fused, and coarsened during the homogenization process. The primary \( \alpha – \text{Cu} \) phase of the semisolid slurry is between a stable and unstable state during the isothermal treatment process (figure 9). That is to say, it shows that the melting and Ostwald ripening occurs simultaneously in the semisolid slurry [30]. The optimal short-time isothermal treatment parameters are soaking time 15 s to 20 s at 990 °C based on the above analysis.

The coarsening behavior of grains can be studied using the classic Lifshize-Slovitze-Wagner (LSW) theory [38, 39], the coarsening kinetics of primary \( \alpha – \text{Cu} \) phase is described as follows equation (9):

\[
D_t^n - D_{t_0}^n = Kt
\]

(9)

Where \( D_t \) is mean grain size at soaking time \( t \), \( D_{t_0} \) is mean grain size at soaking time \( t_0 \), \( K \) is grain growth constant, \( n \) is an exponent. The coarsening mechanisms is generally considered to be composed of interfacial reaction-controlled diffusion mechanism (\( n = 2 \)), volume diffusion-controlled diffusion (\( n = 3 \)), and grain boundary diffusion-controlled diffusion (\( n = 4 \)) [40]. The coarsening behavior of semisolid slurry during the isothermal treatment tends to be controlled by volume diffusion (\( n = 3 \)) according to the previous experimental research [10, 30, 41].

For the purpose of calculating the coarsening kinetics of primary \( \alpha – \text{Cu} \) phase of CuSn10P1 alloy during isothermal treatment, figure 11 shows the relationship of cubed mean grain size versus soaking time. The coarsening rate coefficient \( K = 4436 \mu \text{m}^3 \text{s}^{-1} \) and regression coefficient \( R^2 = 0.938 \) It is found that the
experimental data shows a reasonable fitting with the LSW equation at $n = 3$, indicating that cubic kinetic is still adapted to coexist with melting mechanism and coarsening mechanism.

4. Conclusions

In this work, the effects of soaking time on microstructure characteristics, element distribution, grain stability, and coarsening behavior of semisolid CuSn10P1 alloy were studied. Some conclusions are as follows:
1. Soaking time affects the mean size, roundness, and microstructure uniformity of primary $\alpha$-Cu phase, and can also improve Tin segregation to a certain extent. The optimal short-time isothermal treatment parameters for the CuSn10P1 alloy is soaking time 15 s to 20 s at 990 °C.

2. The mean size of primary $\alpha$-Cu phase gradually increases and the roundness shows a wavy trend with soaking time prolonged.

3. The primary $\alpha$-Cu phase is unstable during short-time isothermal treatment due to the coexistence of melting mechanism and coarsening mechanism.

Acknowledgments

The authors acknowledge funding for this research from the National Natural Science Foundation of China (51765026) (51665024) (51965028) and China Postdoctoral Science Foundation (2020M673588XB). This work is supported by the National and Local Joint Engineering Laboratory of Advanced Metal Solidification Forming and Equipment Technology, Kunming University of Science and Technology, Kunming, China.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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