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Effect of Composition, High Magnetic Field and Solidification Parameters on Eutectic Morphology in Cu-Ag Alloys

Eutectic Modification by HMF and Solidification

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Abstract: High magnetic field (HMF) and solidification processes were changed during the solidification of both Cu-28 mass %Ag and Cu-72 mass %Ag alloys. The results indicated that the eutectic morphology in Cu-Ag alloys was affected by HMF, composition and solidification parameters. The lamellar spacing of Cu-28 mass %Ag alloy solidified by furnace-cooling was refined by the application of HMF owing to the decreased diffusion coefficient in mushy zone. The lamellar spacing in both Cu-28 mass %Ag sample held at the eutectic temperature and Cu-72 mass %Ag sample by slow controlling cooling was increased by HMF, which might be attributed to the dominated thermoelectromagnetic convection. The lamellar spacing in Cu-72 mass %Ag alloys was increased compared with that of Cu-28 mass %Ag alloys because of the decreased growth rates. In Cu-28 mass %Ag alloy, however, fluid transverse velocity gradient was dominate rather than the growth rate and the imposition of HMF had reverse influences on the lamellar spacing. The lamellar-rod transition of Cu phase was promoted by HMF because of the increased Cu volume fraction in eutectic component. These results shed light on the dependence of eutectic morphology in Cu-Ag alloys on composition, external high magnetic field and solidification parameters.

Keywords: Cu-Ag alloy, eutectic, lamellar spacing, solidification, high magnetic field

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Introduction

Cu-Ag material has a wide application in both DC and pulsed high-field magnets as conductors for winding coils because of the combination of high strength and high electrical conductivity [1–4]. The high strength is required to withstand strong Lorentz forces during the operation of high field magnets. Meanwhile, the outstanding electrical conductivity is needed to minimize the Joule heating. Sakai et al. [1] have reported Cu-Ag composites with high strength of 1,000 MPa and high conductivity of 80 % IACS. An ultimate tensile strength (UTS) of 1,050 MPa and a conductivity of 75 % IACS (IACS = the International Annealed Copper Standard, and 100 % IACS = $1.7241 \mu\Omega \cdot \text{cm}$) were realized in Cu-24 mass %Ag sheets [4]. The strength and conductivity of Cu-Ag alloys are related to the microstructure of the composites, and the initial microstructure plays an important role in determining the final microstructure due to the hereditary of microstructure between the as-cast alloy and the deformed composite [5–9]. In Cu-Ag alloys, the strength increased and the electrical conductivity decreased with increasing Ag composition up to 72 mass% [1]. Cu-Ag alloy with about 24–30 mass % Ag is one of the most promising candidates because of the excellent combination of strength and conductivity. This kind of Cu-Ag alloy is hypoeutectic, and the composites contain two components: proeutectic Cu matrix embedded with Ag precipitates and eutectic consisting of Cu and Ag phases [7, 10–13].

The application of a steady magnetic field is one of the effective methods of controlling the microstructure of the alloys during the solidification [14–20]. An internal electric current naturally arises at a liquid–solid interface owing to the Seebeck effect [21]. The thermoelectric

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current at the presence of an external magnetic field caused fluid motion within the mushy zone and enhanced the convective phenomena [17]. Moreau et al. [22] investigated the effect of a 0.55 T magnetic field on the dendrites of Bi-40 mass %Sn and found that the dendrites are more developed than those without a magnetic field. Lehmann et al. [23, 24] have studied the influence of convection on the mushy zone during the horizontal directional solidification of a Cu-60 mass %Ag and an Al-10 mass %Cu and found that the magnetic field modified the fluid flow in the dendritic network by thermoelectric magnetohydrodynamic (TEMHD) effect. Zuo et al. [6, 7, 25] have previously investigated the influences of the 12-T high magnetic field (HMF) on Cu-Ag alloy and Fe-49%Sn alloy. Moreover, the influences of the magnetic field on the eutectic structure have been reported in details [13]. Li et al. [26] and Liu et al. [27] found that the application of a high magnetic field during the solidification decreased the eutectic spacing of the directionally solidified Al-Al₂Cu eutectic alloy at a low growth speed and the lamellar spacing of eutectics of Al-Si alloys. They thought that was as a result of the decreased diffusion coefficient induced by the external magnetic field. Li et al. [28, 29] found the eutectic lamellar spacing of Cu-Ag alloy was also decreased after applying a 12-T magnetic field. Li et al. [30], however, found the application of static magnetic field increased the lamellar spacing of semi-continuous casting Al-Fe alloys due to the initiation of the secondary arms. Therefore, the effect of the external magnetic field on the eutectic lamellar spacing was not entirely clear and the properties of the materials solidified with a magnetic field are rarely studied.

Cu-28 mass %Ag alloys were solidified under a HMF and two different solidification processings were used in our previous researches [13, 29]. The results showed that the application of HMF had an influence on the microstructure of proeutectic Cu dendrites, Ag precipitates in Cu matrix, and eutectic component. The influence mechanism of both Cu dendrites and Ag precipitates had been reported in previous work. The mechanism of eutectic component, however, has not been mentioned. In order to clarify the influence of the external HMF on the microstructure of eutectic. The Cu-72 mass %Ag (eutectic composition of Cu-Ag system) alloys were also solidified with and without a 12 T magnetic field in this paper.

Experimental

Cu-28 mass %Ag and Cu-72 mass %Ag master alloys were casted with high-purity Ag bars (99.996%) and

oxygen-free Cu bars (99.97%) in an intermediate-frequency vacuum induction furnace under high-purity Ar atmosphere. A sample (9 mm in diameter and 65 mm in length) was machined from the master alloy and was placed in a high-purity graphite crucible. They were sealed with a quartz tube which was evacuated to the pressure of 6.6×10^{-3} Pa and then inflated with high-purity Ar. The sealed sample was located in a vacuum resistance furnace surrounded by a superconducting magnet up to 12 T. The solidification profiles of Cu-28 mass %Ag sample and Cu-72 mass %Ag sample were shown in Figure 1. Two different solidification routes were subjected to fabricate the two kinds of samples, which was capable of clarifying the effect of solidification profiles on microstructure of Cu-Ag alloy. Moreover, 0 T and 12 T HMF were subjected during solidification to investigate the influence of HMF on the solidification structure of the two alloys. The detailed experimental information can be found in previous references [7, 13, 31]. The four kinds of alloys were indicated by CA1 (Cu-72 mass %Ag alloy, with the cooling rate of 10 K/min), and CA2 (Cu-72 mass %Ag alloy, with the cooling rate of 1 K/min), CA3 (Cu-28 mass %Ag alloy, without holding at the eutectic temperature), CA4 (Cu-28 mass %Ag alloy, holding at the eutectic temperature).

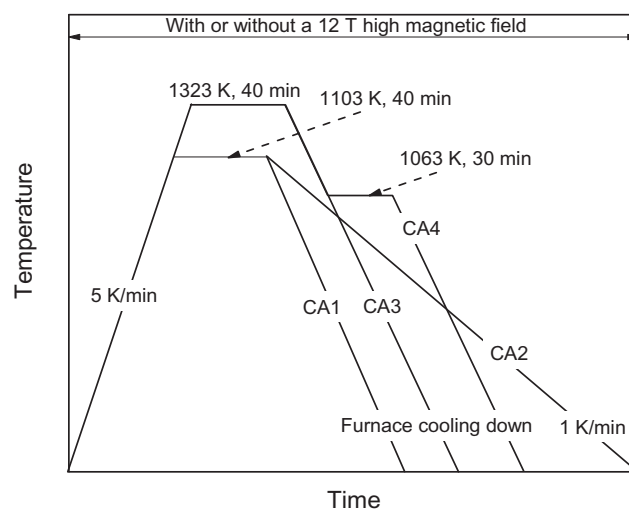


Figure 1: The schematic diagram of solidification routes for Cu-Ag alloys with and without HMF.

The solidified samples were sectioned, polished and etched in a solution of FeCl₃, HCl and C₂H₅OH. The microstructures were observed by SSX-550 scanning electron microscopy (SEM) and analyzed quantitatively by the linear intercept method.

Results

Eutectic spacing and aspect ratio of Cu in Cu-72 mass %Ag alloy

The solidified Cu-72 mass %Ag alloys are primarily composed of eutectics, where the dark contrasts are Cu phases, and the light ones are Ag phases (Figure 2). Some primary Ag dendrites were also found, in which Cu precipitates were found (as shown in the inset in Figure 2). We did not focus on this component because of the low volume fraction and had negligible impacts on properties of alloys. Eutectic Cu is almost lamellar and they are regularly distributed in solidified samples without a HMF (Figure 2(a) and (c)). In the samples processed at a 12 T HMF, however, the volume fraction of the regular lamellar eutectic colonies is decreased and the rod-like Cu phase is increased (Figure 2(b) and (d)).

The inter-lamellar spacing and the aspect ratio were statistically analyzed on more than 20 micrographs and the results are shown in Figure 3. In the case of CA1, the lamellar spacing has no significantly change after the application of 12 T HMF (Figure 2(b) compared by Figure 2(a), and data are shown in Figure 3(a)). In CA2, however, the lamellar spacing increased by 25 % with

the application of 12 T HMF (Figure 2(d) compared by Figure 2(c)). More rod-like Cu phase was found in the 12 T HMF sample, as shown in Figure 3(b). HMF decreases the aspect ratio of eutectic Cu by 30 % and 16 % in CA1 and CA2, respectively. In the samples without HMF, the volume fraction of Ag phase in eutectic component was 64 % and 66 % in CA1 and CA2, respectively. In the samples with 12 T HMF, however, the volume fraction of Ag phase decreased to about 59 % in both CA1 and CA2, and more Ag was existed as primary Ag dendrites. The theoretical volume fraction of Ag phase is 78.76 % using the lever rule. The Cu lamellar spacing in CA2 was larger than that in CA1 regardless with or without HMF.

Microstructural evolution of eutectic region in Cu-28 mass %Ag alloy

The microstructure of Cu-28 mass %Ag alloys (CA3 and CA4) consisted of two components: a Cu-rich proeutectic dendrite (shown as dark contrast in Figure 4), and a eutectic component (shown as light contrast in Figure 4), which formed a network surrounding the Cu-rich dendrites. A rich-Ag layer was seen at the edge of eutectic. The introduction of external HMF during solidification was incapable of changing the shape of the network.

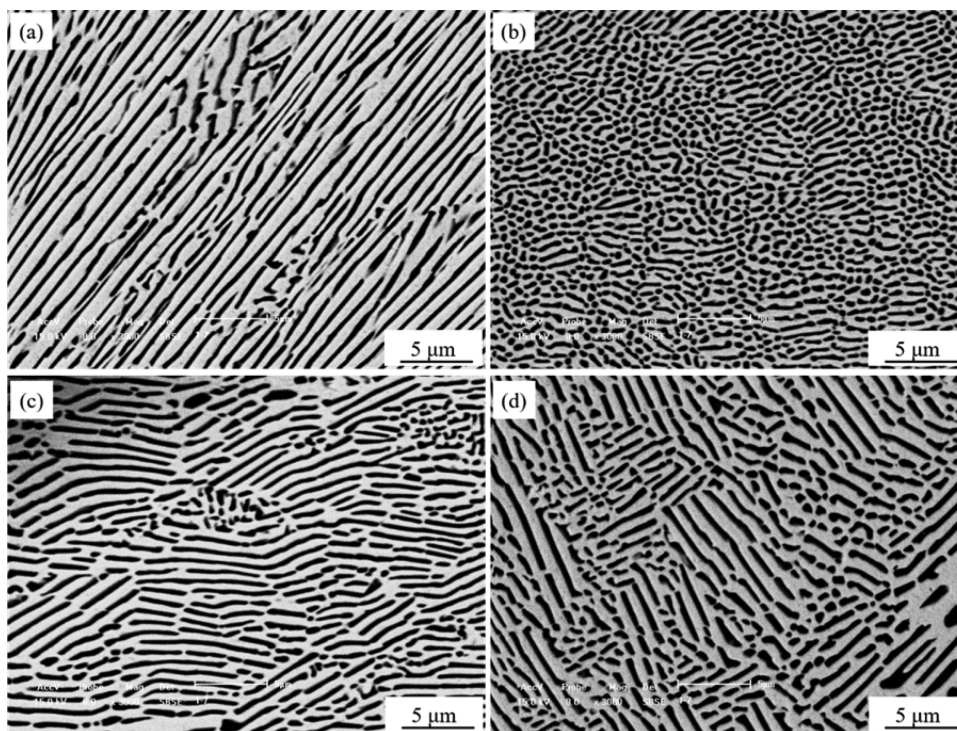


Figure 2: The microstructure of Cu-72 %Ag alloy solidified with different HMFs and solidification profiles, (a) CA1, 0 T, (b) CA1, 12 T, (c) CA2, 0 T, (d) CA2, 12 T. The inset in (a) showed the morphology of primary Ag phase.

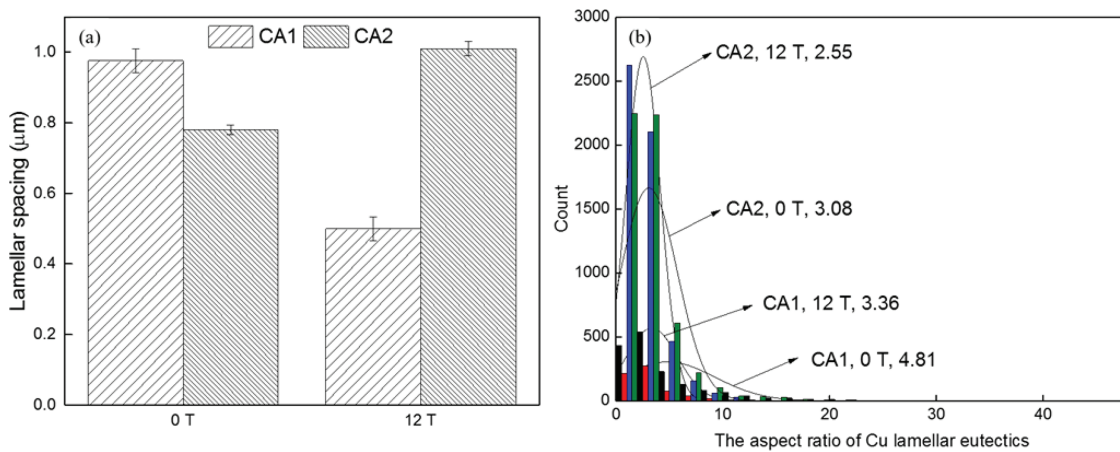


Figure 3: (a) The lamellar spacing and (b) the aspect ratio of Cu lamellar eutectics in Cu-72 %Ag alloy solidified without and with a 12 T HMF.

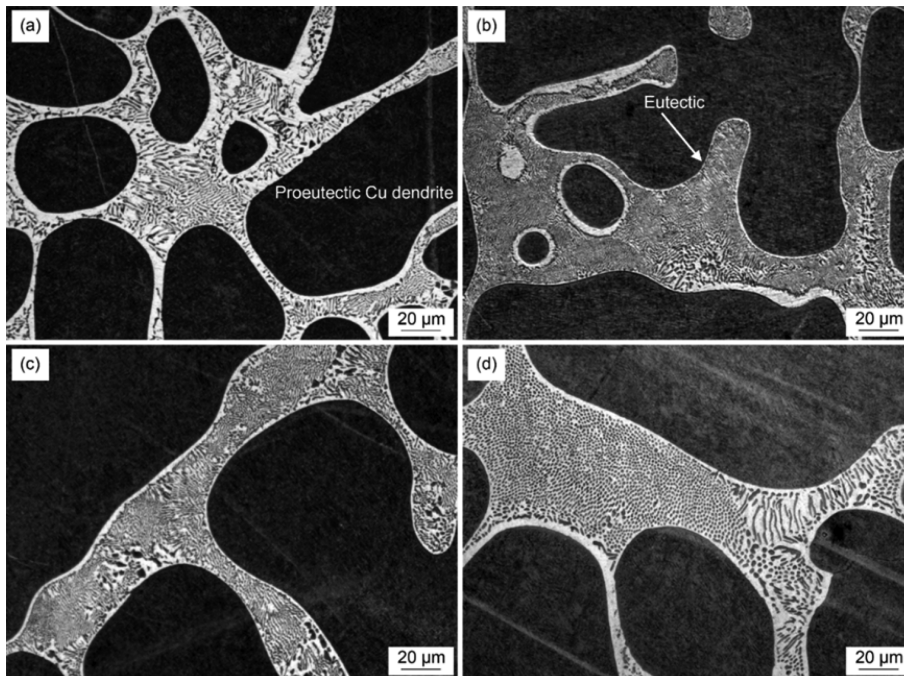


Figure 4: The longitudinal microstructure of Cu-28 mass %Ag alloy with different magnetic condition, (a) CA3, 0 T, (b) CA3, 12 T (c) CA4, 0 T, (d) CA4, 12 T.

In the case of CA3, HMF decreased both the lamellar spacing of eutectic component and the thickness of Ag-rich layer, as shown in Figure 5(a) and (b). In CA4, however, HMF has a reverse impact on the average lamellar spacing and the thickness of Ag-rich layer compared with case CA3 (Figure 5(c) and (d)). In the samples without HMF, the lamellar spacing in eutectic component in CA4 was smaller than that in CA3. In the samples with 12 T HMF, however, the lamellar spacing in CA4 was larger than that in CA3. The results showed that both solidification process and HMF condition could change the microstructure of eutectic component in Cu-28 mass %Ag, and the variation tendency depend on the solidification

process and HMF condition in Cu-28 mass %Ag alloy was the same with that in Cu-72 mass %Ag alloy.

Discussion

The influence of HMF on the volume fraction of Ag phase in eutectic component

Li et al. [32] evaluated the maximum value of $(T_M - T_0)_{\max}$, where T_M and T_0 were the solidification temperatures

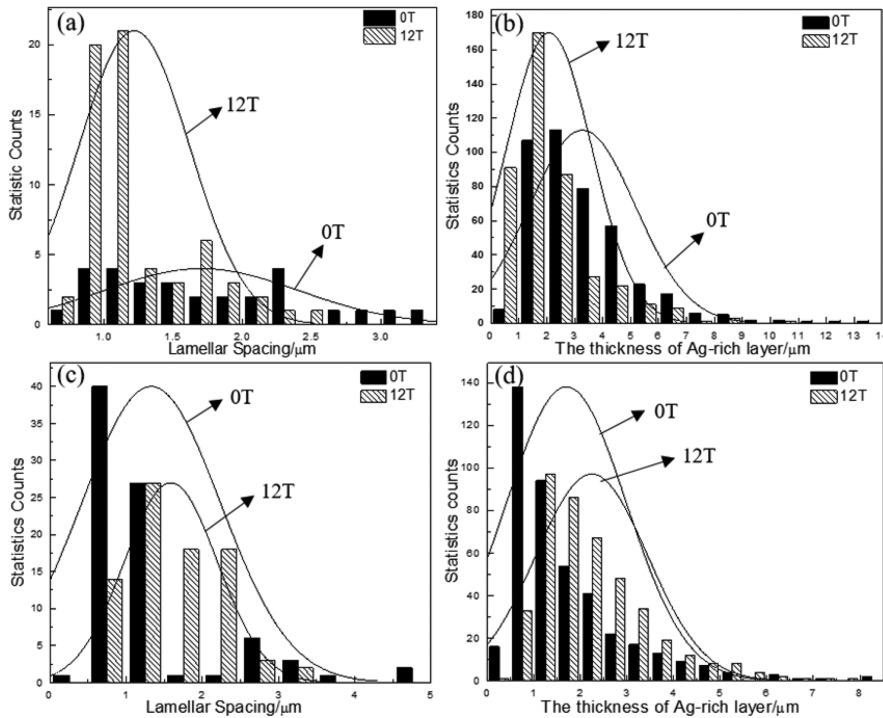


Figure 5: The statistic results of the lamellar spacing (a) and the thickness of Ag-rich layer (b) of CA3, and the lamellar spacing (c) and the thickness of Ag-rich layer (d) of CA4 without and with a 12 T HMF.

with and without the magnetic field, respectively. They found that the HMF had a negligible effect on the eutectic points of non-ferromagnetic alloys, but increased the nucleation undercooling. Both Cu and Ag are non-ferromagnetic substances. Primary Ag phases were found in both without and with HMF samples, so the original composition of alloy was a little higher than the eutectic composition (C_0 in Figure 6). According to the lever rule, the volume fraction of primary Ag phases can be calculated as (AB/AC) . Owing to the increased undercooling of eutectic under the magnetic field, the volume fraction of primary Ag phases increased to $(A'B'/A'C')$, as shown in Figure 6. As a result, the volume fraction of Ag in eutectic component of 12 T HMF sample was decreased.

The influence of HMF on the aspect ratios of Cu lamellar

The rod-lamellar transition is developed by Jackson and Hunt. It was developed and related to the volume fraction of the minor phase (Cu in Ag-Cu eutectic), f , and the interface energy parameters, S , for rod and lamellar microstructures [33].

$$S = 0.7131f^{-0.2327}(1-f)^{-0.1991} \quad (1)$$

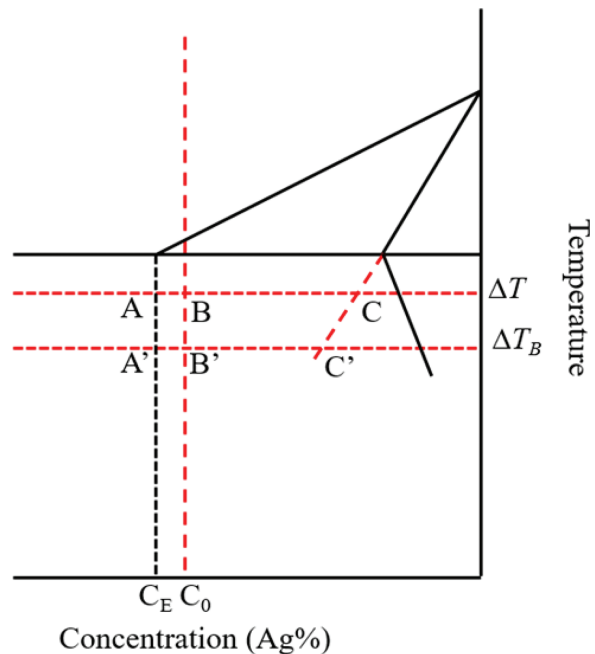


Figure 6: Phase diagram and undercooling (ΔT) of the alloy without and with the HMF.

If the interface energy parameter S is greater than 1.09, the growth morphology will be either purely rod or purely lamellar. Moreover, a larger value of S favors lamellar growth rather than rod eutectic over a smaller volume

fraction of the rod phase [33]. In our case, the fraction of the minor phase f without magnetic field was smaller than that with a 12-T magnetic field. The calculated results show that the S value of 12 T sample is smaller than that of 0 T sample. This caused the enhancement of rod eutectic morphology after the magnetic field was introduced, which was consistent with our experimental results.

The influence of HMF and solidification parameters on the lamellar spacing

Two main mechanisms have been proposed to explain the effect of magnetic field on electrical conductive melt. The first one is the electromagnetic damping force effect (EMDF) which suppresses the natural convection of the melt because of the induced Lorentz force. The suppressed degree of magnetic to the convection can be expressed by the Hartmann number (Ha), which is the ratio of electromagnetic damping forces to the viscous forces. The Hartmann number and can be formulated by $Ha = \sqrt{\sigma/\mu}BL$, where σ is the electrical conductivity, μ is the viscosity of the fluid, L is a typical size of the flow, and B is the magnetic field. Considering the scale of the spacing of eutectic, Ha number is very small imposed by a 12 T magnetic field. Therefore the applied magnetic field does not suppress the convection of the melt during the growth of eutectic. Another effect caused by the magnetic is predominant, which is so-called thermoelectromagnetic convection (TEMC) effect [14, 17, 34]. According to the relationship between the current direction and magnetic field, it can be judged that the new motion is in the transverse sections by the Fleming's rule. Besides, the report shows that the diffusion coefficient decreases as B^{-4} or B^{-2} with the applied magnetic field [35]. As a result, the TEMC effect and decreased diffusion coefficient caused by the applied magnetic field will significantly influence the eutectic growth during the solidification.

The eutectic spacing can be expressed by the formula

$$\lambda^2 = \frac{\pi^2 A k D_L}{m_L (C_{\alpha m} - C_{\beta m}) R} \quad (2)$$

where k is equilibrium distribution coefficient, A is a constant, D_L is the solute diffusion coefficient in the liquid. $C_{\alpha m}$ and $C_{\beta m}$ is the equilibrium solid solubility at the eutectic temperature of α -Cu and β -Ag, respectively. m_L is the slope of liquid line, R is the growth rate. On the other hand, the effect of convection on lamellar spacing for eutectic growth is expressed as [36]:

$$\left(\frac{\lambda}{\lambda_0}\right)^2 = \frac{1}{1 - (2D_L G_u / R^2)} \quad (3)$$

where λ is the lamellar spacing, λ_0 is the lamellar without convection, G_u is the fluid transverse velocity gradient on the solid-liquid interface.

The TEMC can only affect the solutes diffusion on the plane normal to magnetic field. So on the longitudinal sections, the decreased diffusion coefficient by HMF was dominate and decreased the lamellar spacing. According to eq. (2), the lamellar spacing decreases as the diffusion coefficient decreases. As a result, the lamellar spacing of eutectic in CA3 (Cu-28 mass %Ag alloy with furnace cooling) decreased with the application of 12 T HMF. In CA1 (Cu-72 mass %Ag alloy with furnace cooling), however, the decreased diffusion coefficient had no significant influence on the lamellar spacing. One of the reasons might be the existence of Cu dendrites in Cu-28 mass % Ag alloy. In Cu-28 mass %Ag alloy, the Ag phase in eutectic firstly nucleated at the interface of Cu dendrite because of the solute enrichment. The diffusion coefficient has significant influence on the growth of eutectic component. In the Cu-72 mass %Ag alloy, however, the nucleation of eutectic was almost occurred simultaneously. The influence of diffusion coefficient on the lamellar spacing was not as significant as that in Cu-28 mass %Ag alloy. In CA2 (Cu-72 mass %Ag alloy with slow cooling rate) and CA4 (Cu-28 mass %Ag alloy with holding 40 min at 790 °C), however, the action period of HMF was much longer than that in CA1 and CA3. With the increasing action period of HMF, TEMC will be the dominant reason for the change of lamellar spacing. According to the eq. (3), the higher intensity convection G_u lead to an increase in lamellar spacing. The lamellar spacing increased as increased.

Not only HMF but also the solidification parameters have influences on the lamellar spacing. In Cu-72 mass % Ag alloy, the growth rate (R) in CA2 is smaller than that in CA1 because of the slow cooling rate, which leads a larger lamellar spacing based on eq. (3). In Cu-28 %Ag alloy, G_u in CA4 is smaller than that in CA3 because temperature gradient becomes smaller during the holding process, which caused the decreased lamellar spacing in 0 T samples. With the application of 12 T HMF, however, the TEMC must be considered in CA4 because of the long action period of HMF, which caused the decreased of concentration gradient in front of solid-liquid interface. The growth rate decreased with decreased concentration gradient. As a result, the lamellar spacing in CA4 was larger than that in CA3 when the 12 T HMF was applied.

Conclusions

The influences of a 12 T HMF and solidification process on the microstructure of eutectic component in Cu-28 mass % Ag and Cu-72 mass %Ag alloy were investigated. The volume fraction of Cu phase in eutectic was increased, and the aspect ratio of Cu lamellar was decreased with the application of HMF. The increased volume fraction of Cu phase promoted the transition from lamellar to rod of Cu.

The lamellar spacing of the eutectic is decreased with the application of magnetic field in furnace-cooling Cu-28 mass %Ag samples. It was resulted from the decreased diffusion coefficient after applying the HMF. In the Cu-28 mass %Ag sample held at eutectic temperature and slow-cooling Cu-72 mass %Ag sample, however, the lamellar spacing was increased after the application of HMF. The TEMC effect was dominant because of the longer action period of HMF, which caused the higher intensity convection and increased the spacing. On the other hand, the solidification process changed the growth rate and fluid transvers velocity gradient on the solid–liquid interface, thus changing the lamellar spacing of eutectics.

In summary, the microstructure of eutectic was related to the diffusion of solute, the convection of melt in front of solid–liquid interface, and the growth rate. The refined lamellar spacing of eutectic is as a result of by the combined action of HMF and solidification process.

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