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Effect of uniform magnetic field on crystallization of intermetallic compound layers between Cu and liquid SnZn alloys

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Abstract: Crystallization of intermetallic compound layer between Cu and SnZn alloy under uniform magnetic field was studied. The effect of magnetic field density on the growth behavior of the intermetallic layer such as microstructure, crystal orientation and composition was analyzed by scanning electron microscopy, X-ray diffraction and electron-probe microanalysis, respectively. Compared with the intermetallic layer without magnetic field, 0.1 T of magnetic flux density decreases the layer thickness. However, further increasing magnetic flux density promotes the layer growth. Application of magnetic field also changes the crystal orientation of intermetallic layer, but has no obvious influence on the layer composition. This phenomenon can be attributed to the role of thermo-electromagnetic convection and Lorentz force on the Cu dissolution as well as the accumulation of Cu solute at the interface front.

Key words: magnetic field; SnZn alloy; intermetallic compound; Cu dissolution

1 Introduction

The recent development of superconducting magnetic techniques has provided a rather large opportunity to apply high magnetic field to microstructure control during the solidification processing and phase transformation [1,2]. Many researches about microstructure control under magnetic field are related to the metal crystallization behaviors. The intermetallic compound (IMC) layers, as one of the products from the crystallization between contacting dissimilar metals, have practical significance in such consideration as diffusion bonding and soldering, coating and corrosion [3,4]. Spontaneously, high magnetic flux density as an interesting parameter is paid much attention to the microstructure control of IMC layer in recent years.

Many studies have been conducted to realize the layer crystallization of IMC under high magnetic fields. It has been recognized that high magnetic field can affect the layer growth of IMC by changing the atomic diffusion in the solid−solid couples [5−8]. Once a liquid phase is involved, however, the mass transfer during IMC growth also depends on the liquid convection in the case of liquid−solid couples. Generally, there are two typical effects induced by magnetic field on the liquid convection: the natural liquid convection damped by Lorentz force, and the thermo-electromagnetic convection (TEMC) which origins from the interaction between thermoelectric currents and magnetic field [1]. Although there are several examinations carried out on the IMC growth under the influence of Lorentz force and TEMC, a complete description is not yet available. For example, the application of high magnetic field has a non-linear influence on the IMC layer thickness in Al/Cu and Al/Zn couples [9,10], which has been interpreted from the changed diffusion of liquid Al into the solid by Lorentz force suppressing natural convection and TEMC. However, the solidification of Al−Cu alloy in magnetic field by LI et al [1,11] indicated that TEMC still significantly affected the liquid−solid interface even under a weak magnetic field (≤0.5 T). Therefore, much more work needs to be conducted from low density of magnetic field to high density to identify the role of TEMC and Lorentz force during IMC growth process.

More importantly, the growth kinetics and morphology of IMC change a lot when the formation of

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IMC layer is controlled by the mass transfer from the diffusion and the dissolution of solid substrate into the liquid bath [12−15]. For instance, high magnetic field promoted the IMC growth by Lorentz force inhibiting Cu dissolution in Sn1.5Cu/Cu liquid-solid couples [16]. It was interesting to find that high magnetic field changed the morphology of IMC layer from faceted-type to stick-type at the interface between Cu and liquid Sn after that a small amount of Ni was added into the liquid Sn [17]. This investigation indicated that under magnetic field, only adding a small amount of element might strongly modify the growth behavior of IMC layer. However, the growth mechanism of IMC layer under the interaction between high magnetic field and additive is still out of research. In this work, several liquid−solid couples under uniform magnetic field, namely Sn1Zn/Cu, Sn3Ag1Zn/Cu, Sn9Zn/Cu and Sn9Zn2Cu/Cu, were employed to investigate the influence of TEMC and Lorentz force on the layer crystallization of IMC during growth process between the liquid Sn and solid Cu in the presence of Zn additive.

2 Experimental

Four Sn-based alloys, namely Sn1Zn, Sn3Ag1Zn, Sn9Zn and Sn9Zn2Cu, were used in the current experiments. They were prepared with 99.9% Sn, 99.999% Zn, 99.99% Ag, and 99.9% Cu. Proper amounts of the respective elements were weighed for each alloy and put in a quartz tube. The specimen was placed in a furnace and melted in the protection of pure argon atmosphere at 500 °C for several hours and then cast into rods. Cu foils (90 mm×5 mm×0.1 mm) were cleaned in 5% HCl (volume fraction) solution for a few seconds, rinsed with alcohol, and then dried.

The liquid−solid couples were prepared in the super-conduct magnetic field generator (JMT−100). A heating furnace was located under the high magnetic field. A quartz crucible (*d*18 mm×45 mm) was used as a vessel for liquid bath. A detail experimental process can be referenced in our previous investigation. In general, 20 g of Sn-based alloy for each specimen was melted and stabilized at 280 °C under various magnetic flux densities (0 T, 0.5 T, 2 T, 4 T, and 6 T). And then the Cu substrates were dipped into the molten SnZn alloy and reacted for 180 s. The measured temperature fluctuation during this process was controlled within ±2.5 °C, which was improved to have no obvious effect on the layer growth. After reaction, the Cu substrates were extracted quickly from the liquid bath and quenched into water.

To examine the microstructures of IMC layers, the cross-sectional micrographs and top-view morphologies of IMC layers were examined. The specimens were mounted by resin, polished, and then etched with a solution containing 93% methanol, 5% nitric acid and 2% hydrochloric acid (volume fraction). The cross-sections of IMC layers were observed by using scanning electron microscopy (SEM, JSM−5600LV 15 kV). To observe the IMC morphology, the Sn-based alloys in some couples were removed by using a 5% nitric (volume fraction) solution and ultrasonic cleaning. The IMC morphologies were then examined from top-view of the samples by SEM.

The average thickness of IMC layers was also been measured. The thickness l of IMC layer in one cross-sectional micrograph was evaluated as follows:

$$l=A/w \tag{1}$$

where A and w are the total area and length of IMC layer by using Q500IW image analysis meter, respectively. The average thickness of IMC layer for each specimen was calculated from six different local micrographs by above method to reduce the measurement error. Furthermore, the components of interfacial IMC layers were identified using energy dispersion of X-ray (EDX). The crystal orientation of IMC layer was measured by X-ray diffraction (XRD−6000) with Cu K_α irradiation. And the distribution of the IMC component was investigated by electron-probe microanalysis (EPMA−1600).

3 Results

3.1 Effect of magnetic field on microstructure of IMC layers

Figure 1 shows the cross-sectional micrographs of IMC layers in the Sn1Zn/Cu and Sn9Zn/Cu samples treated at 280 °C for 180 s under various magnetic flux densities, as an example to illustrate the cross-sectional microstructure evolution. It is clear that a non-dense IMC layer appears between the Sn-based alloy and Cu for the Sn1Zn/Cu couple without magnetic field, as shown in Fig. 1(a). This non-dense structure is also observed at 0.1 T magnetic flux density, as shown in Fig. 1(c). After Sn1Zn reacted on Cu substrate in 6 T magnetic flux density, a relative thick IMC layer forms at the interface as shown in Fig. 1(e). The similar non-dense structure can also be found in the case of Sn3Ag1Zn/Cu samples. Compared with Sn1Zn/Cu couples, however, a continuous layer forms at Sn9Zn/Cu interface without magnetic field as shown in Fig. 1(b), which is consistent with the results in many previous reports [15,18,19]. Meantime, the thickness of IMC layer is found to be decreased at 0.1 T magnetic flux density, as shown in Fig. 1(d). An interesting finding is that the thickness is increased after Sn9Zn/Cu is treated at 6 T magnetic flux density as shown in Fig. 1(f). The interfacial IMC formed on Cu with Sn9Zn2Cu is also a continuous layer,

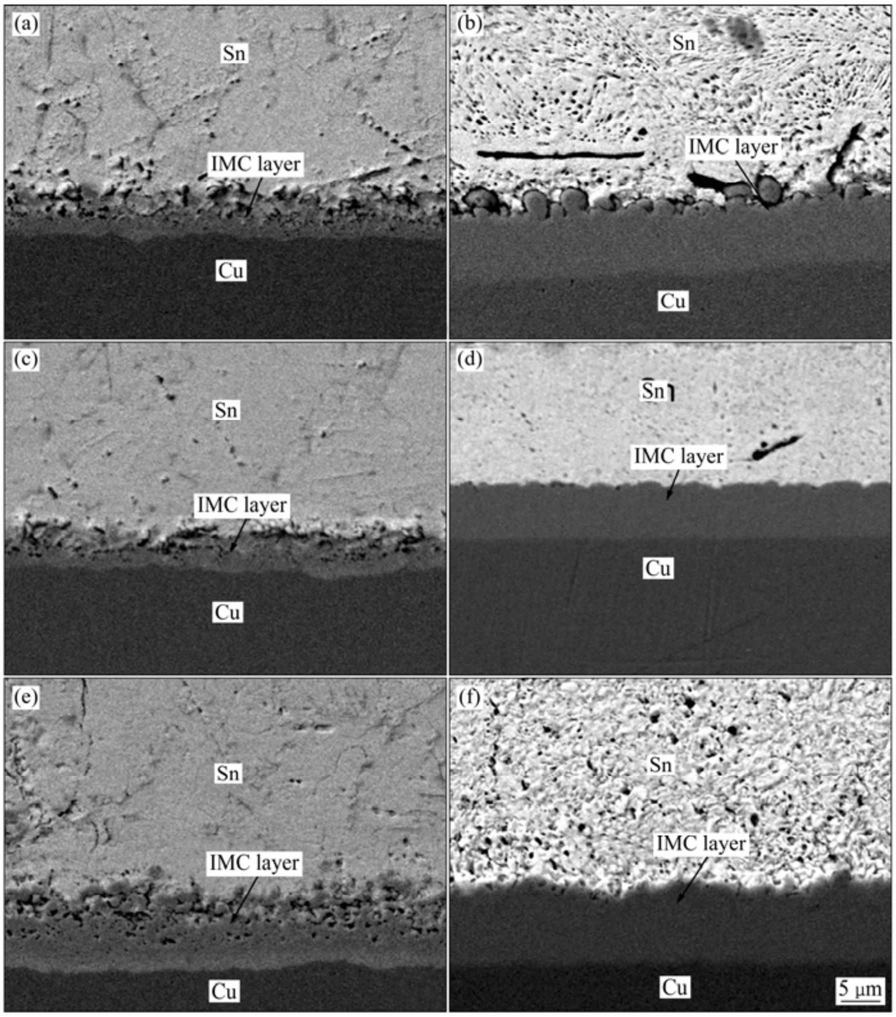

Fig. 1 Cross-sectional micrographs of IMC layers in Sn1Zn/Cu and Sn9Zn/Cu couples treated at 280 °C under various magnetic flux densities: (a) Sn1Zn/Cu, 0 T; (b) Sn9Zn/Cu, 0 T; (c) Sn1Zn/Cu, 0.1 T; (d) Sn9Zn/Cu, 0.1 T; (e) Sn1Zn/Cu, 6 T; (f) Sn9Zn/Cu, 6 T

and the similar result as the Sn9Zn/Cu samples is observed.

Figure 2 shows the top morphology of IMC layer in the Sn1Zn/Cu and Sn9Zn/Cu couples treated at 280 °C for 180 s under various magnetic field conditions. Many small IMC grains are found in Sn1Zn/Cu sample without magnetic flux density, and some small particles are likely to aggregate into large particles, as shown in Fig. 2(a). At 0.1 T magnetic flux density, nearly no aggregation of IMC grains is found as shown in Fig. 2(c), while many coarsened grains present in the micrograph for Sn1Zn/Cu at 6 T magnetic flux density, as shown in Fig. 2(e). The IMC morphology for Sn3Ag1Zn/Cu sample is likely the same as that for Sn1Zn/Cu sample. In the case of Sn9Zn/Cu sample without magnetic field, many large grains resulting from grain coarsening appear, as shown in Fig. 2(b). It is noted that there also exist several cracks in IMC layer shown in Fig. 2(b), which is possibly attributed to the induced stress during the growth process. In contrast, the average IMC grain size for Sn9Zn/Cu at 0.1 T magnetic flux density becomes much smaller than that without magnetic field as indicated in Fig. 2(d), and more cracks develop, as shown in Fig. 2(d). Further increasing magnetic flux density up to 6 T also decreases the average size of IMC grains. However, the development of cracks is obviously inhibited, as indicated in Fig. 2(f).

3.2 Effect of magnetic field on crystal orientation and composition of IMC layers

To identify the phase composition and crystal orientation of interfacial IMC layer, the samples for morphology observation were also analyzed by XRD. Figure 3 presents the typical XRD patterns for IMC layers in the Sn1Zn/Cu and Sn9Zn/Cu samples treated at 280 °C for 180 s under various magnetic flux densities. The XRD patterns reveal a relatively high remarkable intensity of Cu_5Zn_8 (444), (330) and (741) for the Sn1Zn/Cu samples. Compared with the Sn1Zn/Cu samples without magnetic field, magnetic field has no

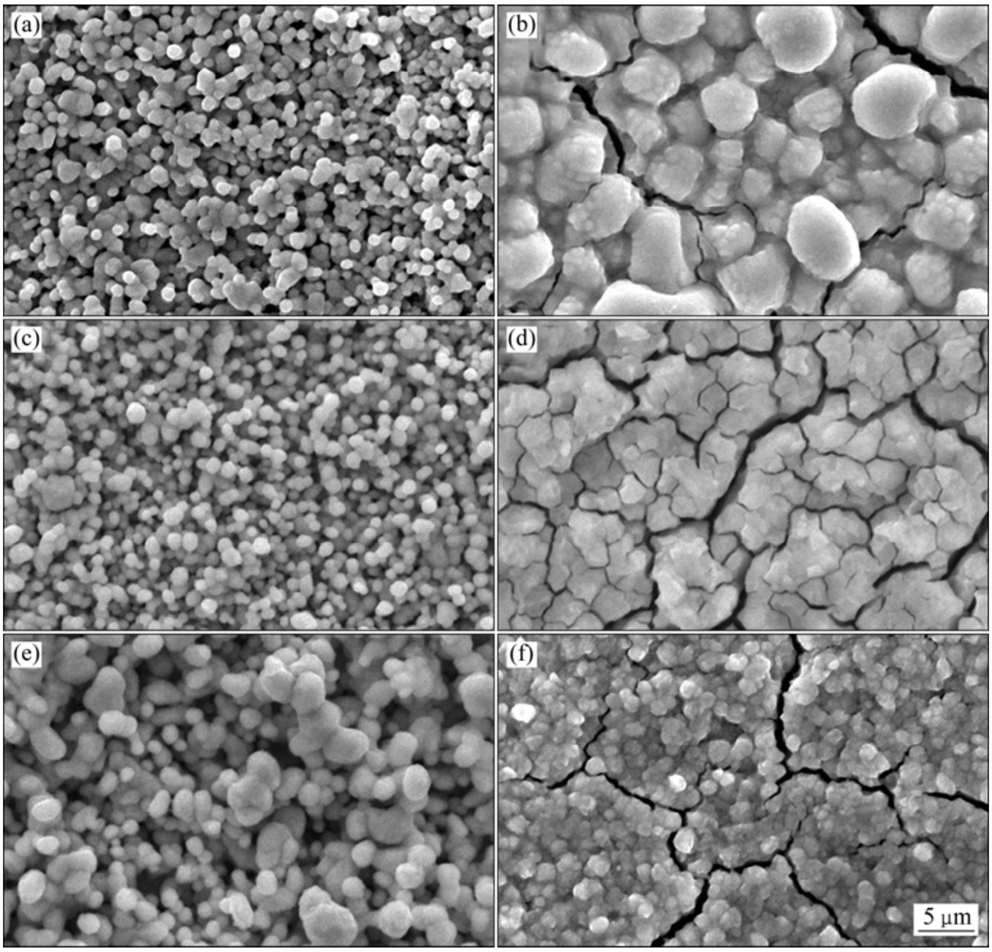

Fig. 2 Top morphologies of IMC layer in Sn1Zn/Cu and Sn9Zn/Cu at 280 °C for 180 s under various magnetic flux densities: (a) Sn1Zn/Cu, 0 T; (b) Sn9Zn/Cu, 0 T; (c) Sn1Zn/Cu, 0.1 T; (d) Sn9Zn/Cu, 0.1 T; (e) Sn1Zn/Cu, 6 T; (f) Sn9Zn/Cu, 6 T

obvious effect on the crystal orientation of Cu_5Zn_8 IMC except the peak intensities. As can be seen, the intensities of all Cu_5Zn_8 peaks at 0.1 T magnetic flux density are much weaker than those without magnetic flux density, while 6 T magnetic flux density increases the IMC intensities, as shown in Fig. 3(a). In the case of Sn9Zn/Cu without magnetic flux density, however, it is found that the three characteristic peaks of Cu_5Zn_8 change into (444), (330), and (222), among which the (330) plane becomes the highest peak, as shown in Fig. 3(b). In 0.1 T magnetic flux density, the highest peak with (444) plane appears, while 6 T magnetic flux density increases the intensity of (330) Cu_5Zn_8, as shown in Fig. 3(b). This suggests that magnetic field can change the crystal orientation of IMC layer.

Figure 4 shows the content profiles of Cu_5Zn_8 components from the Cu side to the Sn side for the Sn1Zn/Cu and Sn9Zn/Cu samples treated at 280 °C for 180 s under various magnetic field conditions. In the case of Sn1Zn/Cu sample without magnetic field, the Cu content decreases from the Cu side to the Sn side while the Sn content gradually increases, as shown in Fig. 4(a). There exists fluctuation for Sn content and the fluctuation becomes more serious in magnetic field, especially for Sn1Zn/Cu at 0.1 T magnetic flux density, as shown in Figs. 4(a), (c), and (e). From Fig. 1 and Fig. 4, the presence of Sn in the IMC layer proves the existence of residual Sn in the incompact layer. High content of accumulated Zn is also found in the IMC layer. Moreover, the measured content of Zn component is lower than the stoichiometric proportion of Cu_5Zn_8 due to the existing residual Sn, as shown in Figs. 4(a), (c), and (e). On contrast for the Sn9Zn/Cu sample without magnetic field, it is apparent that the exact stoichiometric proportion of Cu_5Zn_8 can be found and nearly no Sn can be observed in the IMC layer, as shown in Fig. 4(b). Magnetic field has also no obvious effect on the composition of IMC layer in Sn9Zn/Cu samples, as shown in Figs. 4(d) and (f).

3.3 Effect of magnetic field on growth thickness of IMC layers

Figure 5 shows the thickness evolution of IMC under the influence of magnetic field for Sn1Zn/Cu,

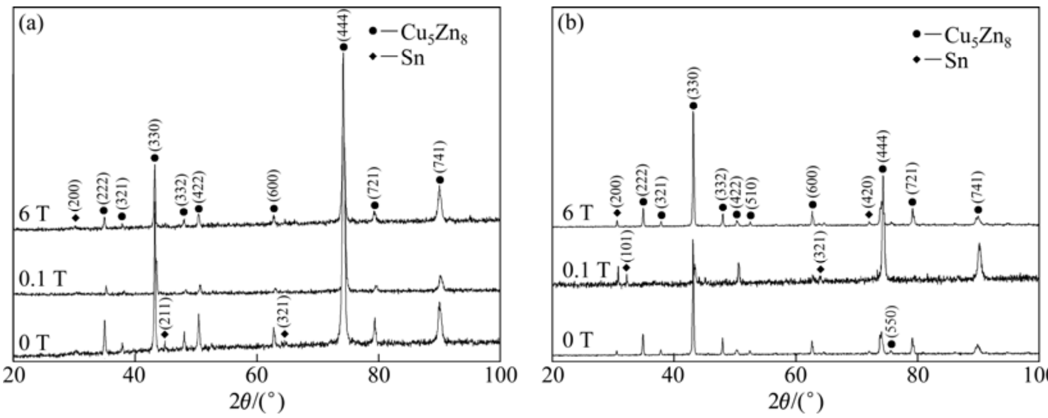

Fig. 3 XRD patterns for IMC layers in Sn1Zn/Cu (a) and Sn9Zn/Cu (b) samples treated at 280 °C for 180 s under various magnetic flux densities

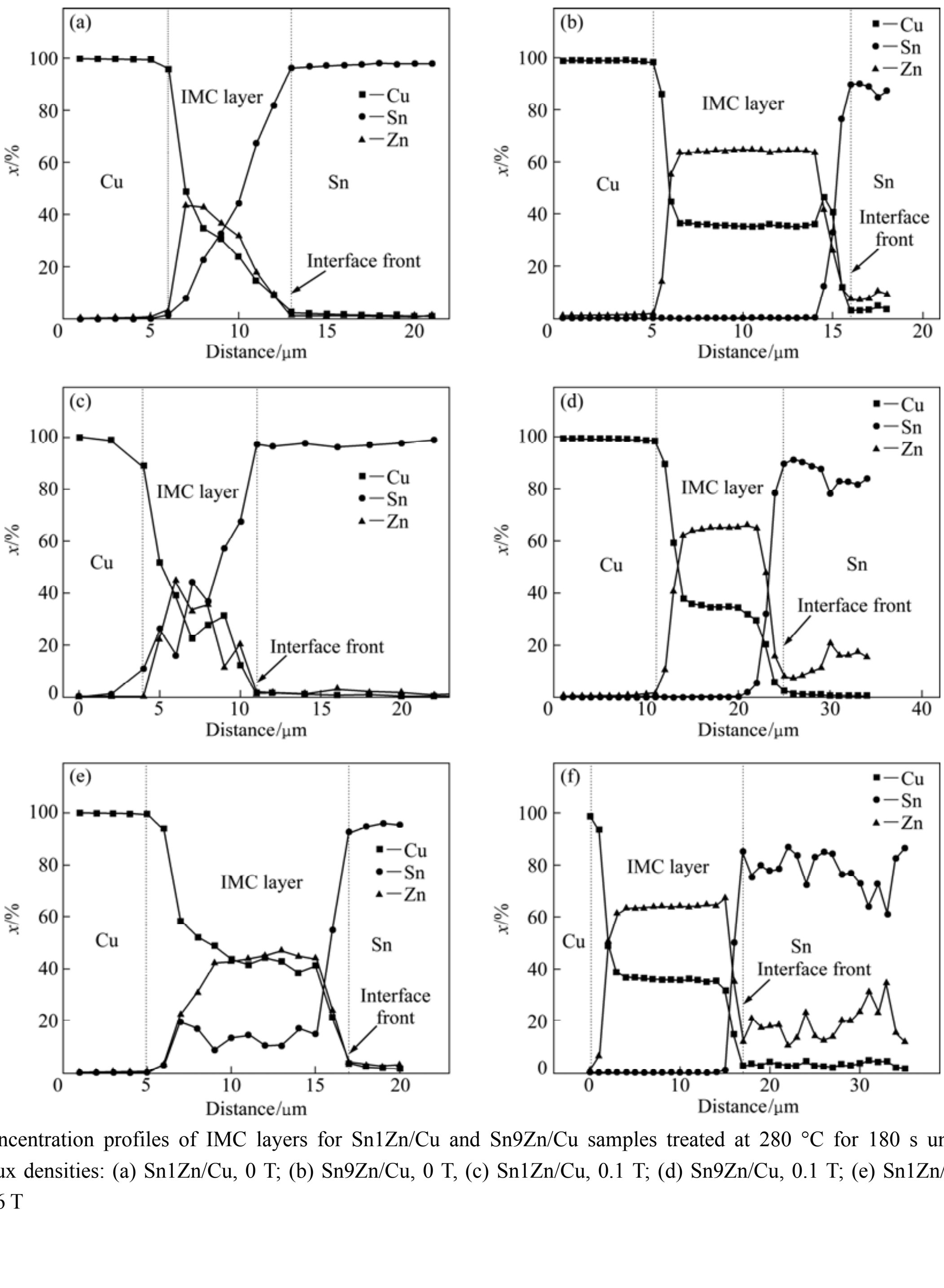

Fig. 4 Concentration profiles of IMC layers for Sn1Zn/Cu and Sn9Zn/Cu samples treated at 280 °C for 180 s under various magnetic flux densities: (a) Sn1Zn/Cu, 0 T; (b) Sn9Zn/Cu, 0 T, (c) Sn1Zn/Cu, 0.1 T; (d) Sn9Zn/Cu, 0.1 T; (e) Sn1Zn/Cu, 6 T; (f) Sn9Zn/Cu, 6 T

Sn3Ag1Zn/Cu, Sn9Zn/Cu, and Sn9Zn2Cu/Cu after being treated at 280 °C for 180 s. It is found that the measured thickness of IMC layer in Sn9Zn/Cu or Sn9Zn2Cu/Cu sample is much thicker than that in Sn1Zn/Cu or Sn3Ag1Zn/Cu samples because more active Zn is available for the crystallization of Cu_5Zn_8. Compared with the layer thickness without magnetic field, it is interesting to see that the measured thickness of IMC layer is decreased at 0.1 T magnetic flux density, as indicated in Fig. 5. However, the thickness increases with further increasing the magnetic flux density up to 6 T, which is consistent with our previous result [16]. This suggests that magnetic field densities have non-linear effect on the layer growth of IMC between the solid Cu and liquid Sn with Zn additive, where the low density can probably suppress the IMC growth.

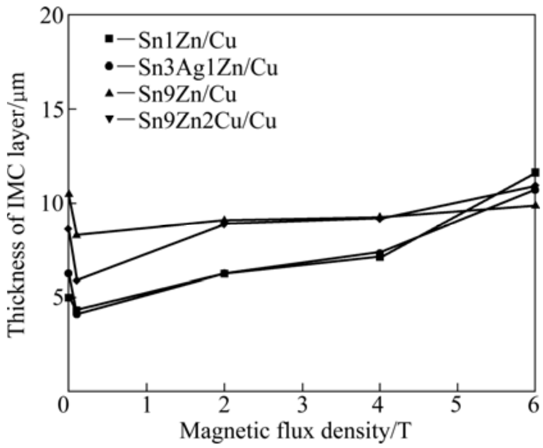

Fig. 5 Thickness of IMC layers in Sn1Zn/Cu, Sn3Ag1Zn/Cu, Sn9Zn/Cu and Sn9Zn2Cu/Cu treated at 280 °C for 180 s under various magnetic flux densities

4 Discussion

The experimental results indicate that the applied magnetic field has changed the microstructure of IMC layer for Sn1Zn/Cu, Sn3Ag1Zn/Cu, Sn9Zn/Cu and Sn9Zn2Cu/Cu samples, while it has no obvious influence on the IMC composition. Most importantly, the results also imply that magnetic flux density has no-linear effect on the IMC layer thickness. In general, the growth of most IMC layer between Cu and liquid Sn alloy is mainly affected by the atomic diffusion in the IMC layer as well as the Cu dissolution into the liquid bath [14,15]. From the view point of Cu, when Cu foil comes into contact with molten Sn-alloy it starts to dissolve rapidly. The dissolution flux of all involved Cu from Cu substrate is basically composted of two parts: the one is the flux for IMC growth at the interface and the other is the flux of Cu dissolved into the liquid bath [14−16]. Decreasing the flux of Cu into the liquid might promote the flux for IMC layer growth and thus increase the IMC thickness. For example, adding Cu into molten Sn can suppress the Cu dissolution and increase the thickness of Cu_6Sn_5 IMC [15,20]. However, the liquid convection at the interface front between IMC layer and liquid Sn directly affects the mass transport process. Stronger convection currents at the interface front promote the mobility of dissolved Cu solute into the liquid bath and then probably decrease the Cu flux for IMC formation. Under the magnetic field, both TEMC and Lorentz force can modify the liquid convection at the interface front.

When the liquid metal moves around the interface front, two types of electric currents may be induced in magnetic field: the first one is the current generated by the liquid motion across the magnetic field lines, and the second one is the thermoelectric current which results from the Seeback effect by the temperature gradient at the interface front between IMC and liquid. And the electric current density relates to these two currents which can be represented as [1]:

$$\boldsymbol{J} = \sigma \boldsymbol{u} \times \boldsymbol{B} + \sigma S \nabla T \tag{2}$$

where $\boldsymbol{J}$ is the electric current density; σ, S, $\boldsymbol{u}$ and $\boldsymbol{B}$ respectively denote the electrical conductivity, the absolute thermoelectric power of the conducting medium, the liquid velocity field and the applied magnetic field. The first item at the right side in Eq. (2) represents the induced electric current generated by the liquid motion itself, and the second item is the thermoelectric effect in the presence of a temperature gradient. Under the applied magnetic field, the interaction between the electric current and magnetic field produces the force $\boldsymbol{F}$ which can change the liquid motion [1]:

$$\boldsymbol{F} = \sigma(\boldsymbol{u} \times \boldsymbol{B}) \times \boldsymbol{B} + \sigma S \nabla T \times \boldsymbol{B} \tag{3}$$

The first item at the right side of Eq. (3) shows the Lorentz force induced by the interaction between liquid motion and magnetic field. The produced Lorentz force usually inhibits the liquid convection and many researchers have applied Lorentz force to braking the liquid metal [21,22]. Our previous study shows that high magnetic field has retarded the dissolution of Cu and thus enhanced the layer growth of IMC [16]. In the present work, the retarding effect of Lorentz force on liquid convection is supposed to be a main reason for the increase of the IMC layer thickness with the density increasing from 1 T to 6 T. However, at the interface front between IMC and liquid Sn, a temperature gradient always produces electric current because of the thermoelectric effect and the interaction between thermoelectric current and magnetic field can generate the driving force for a new convection, namely TEMC, as indicated by the second item at the right side of Eq. (3). The roles of Lorentz force and TEMC on the interface shape and product morphology during the directional solidification of Al−Cu alloy have been theoretically analyzed by LI et al [1]. It can be realized from their work that the TEMC effect on the interface morphology becomes significant when a weak magnetic flux density (≤0.5 T) is applied. In the present case of IMC layer between SnZn alloy and Cu at 0.1 T magnetic flux density, it is possible that the TEMC decreases the flux of Cu for IMC formation by promoting the dissolution of Cu into liquid bath and thus suppresses the layer growth of IMC. The influence of Lorentz force and TEMC on dissolution flux of Cu into the liquid bath can be reflected by the content of Cu solute at the interface front, because Cu solute possibly accumulates at the interface front under the condition of a weak convection and small Cu dissolution flux. Therefore, the average content of Cu solute at the interface front was carefully calculated from the data in Fig. 4. Table 1 shows the average content of Cu solute at the interface front between IMC layer and liquid Sn in Sn1Zn/Cu and Sn9Zn2Cu/Cu samples. Indeed, the content of Cu solute at 0.1 T magnetic flux density is less than that without magnetic field, and then it increases when the applied magnetic flux density is increased up to 6 T. The non-monotonic growth of IMC layer resulting from the application of magnetic field was also reported on Al/Cu couples by LI et al [9]. Since the growth of IMC layer in Al/Cu couple was controlled by the diffusion of Al atoms from liquid into the IMC layer, the influence of TEMC resulted in the increase of IMC layer thickness while Lorentz force retarding liquid convection inhibited the IMC growth. In contrast to the cited work [9], it is noted that the growth of IMC layer between Cu and Sn-based alloy is mainly affected by Cu dissolution from solid substrate to the liquid and IMC precipitation. And thus different roles of Lorentz force and TEMC due to the variation of IMC growth mechanism were observed during the crystallization of IMC layer in the present work.

Table 1 Average content of Cu solute at interface front in Sn1Zn/Cu and Sn9Zn/Cu samples treated at 280 °C for 180 s under various magnetic flux densities

Sample	x/%		
	0 T	0.1 T	6 T
Sn1Zn/Cu	1.60	0.81	2.30
Sn9Zn/Cu	4.96	1.52	3.21

5 Conclusions

1) The IMC layer formed in Sn1Zn/Cu and Sn3Ag1Zn/Cu is an un-compact layer, and relatively thick and dense layer forms in Sn9Zn/Cu and Sn9Zn2Cu/Cu samples. Application of uniform magnetic field has non-linear effect on the average size of IMC grains.

2) The crystal orientation and composition analysis reveals that the interfacial IMC is composed of Cu_5Zn_8. Both 0.1 T and 6 T magnetic flux densities change the crystal orientation of interfacial IMC, but have no influence on the composition of IMC layer.

3) From the growth kinetics of IMC layer, compared with the IMC without magnetic field, application of 0.1 T magnetic flux density retards the IMC growth. Nevertheless, the IMC growth increases as magnetic flux density increases further. This non-linear growth of IMC layer is probably attributed to the intrinsic effect of thermo-electromagnetic convection and Lorentz force on the Cu dissolution and the accumulation of Cu solute at the interface front.

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均恒磁场对 Cu 与液态 SnZn 合金间化合物层结晶行为的影响

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摘　要：以 Cu 和 SnZn 合金的金属间化合物界面结晶为对象，研究均恒强磁场对界面化合物界面行为的影响，并通过扫描电镜、X 射线衍射及电子探针分析磁场对界面金属间化合物组织、相结构及成分的影响规律。结果表明：与无磁场下的界面金属间化合物相比，0.1 T 磁感应强度降低了化合物层的生长，继续增加磁感应强度使得化合物层的生长加快。磁场改变了化合物层的晶体取向，但是对化合物的成分没有明显影响。这是因为磁场下热电磁对流和洛伦兹力对 Cu 溶解及液固界面前沿 Cu 溶质有富集的作用。

关键词：磁场；SnZn 合金；金属间化合物；Cu 溶解

(Edited by LI Xiang-qun)