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ARTICLE

Grain Refinement and Mechanical Properties Improvement of Al-Cu-Ti Alloys After Different Melt Heat Treatment Processes

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Abstract: The modification of Al-Cu alloy by adding grain refiner combined with different heat treatments (superheat treatment and thermal rate treatment) was investigated. The phase composition, solidification microstructure and mechanical properties of Al-Cu-Ti alloys were analyzed. Moreover, the melt structure transformation of Al-Cu-Ti alloys after different melt heat treatments was studied by DSC. The results show that the thermal rate treatment greatly refines the grain of Al-Cu-Ti alloy and improves the mechanical properties. Through the analysis of thermodynamic phase transition, it is found that the latent heat of fusion of Al-Cu-Ti alloy after superheat treatment and thermal rate treatment becomes smaller with the increase of interfacial energy, which refines the microstructure of the alloy to some extent.

Key words: melt heat treatment process; melt structure; solidification structure; grain refinement; mechanical properties

The solidification of alloys originates from the melt and terminates in the solid, which delivers the information of liquid-solid phase transformation[1]. As the melt of alloys is the parent state of solid alloy, the structure and properties of melt could affect the solidification microstructure and thus influence the properties of alloys. Therefore, the alloy with the same composition after different melt treatments may have different solidification microstructure and morphology characteristics[2,3]. The physical or chemical treatment can change the structure of alloy melt, and affect its solidification and crystallization state, thus achieving the purpose of "controlling structures" and "controlling properties"[4]. However, it is difficult to directly investigate the liquid alloy at high temperature. There are few studies on the high-temperature melt structure due to the structural complexity of alloy melts. To this end, people developed an amorphous model, a hole model, a microcrystal model, a dislocation model and a comprehensive model, etc, to qualitatively characterize the characteristics of alloy melt structure whose superheat is not very large. Some experimental mechanisms based on the model above were established. However, these theories have a certain limitation and lack systematicity[5]. The structural or state changes could occur when the alloy undergoes melt superheating treatment, thermal-cold cycling treatment, or aging treatment. Correspondingly, the physical properties of alloy melt such as electrical resistivity and viscosity could change. At the same time, the solidification microstructure also has different changes leading to different mechanical properties.

As one of the heat-resistant aluminum alloys with high-strength and excellent durability, Al-Cu systems are widely used in light-weight constructions and transport applications. In recent study, research found that Al-Cu master alloy reinforced with titanium diboride can be prepared by salts route reactions[6]. However, the Al-Cu alloy has a wide crystallization temperature range and poor fluidity, which is liable to cause defects such as shrinkage porosity and thermal cracking, thus reducing the mechanical properties and limiting the application[7-9]. A large number of studies have found that grain refinement can improve the strength and plasticity of aluminium alloy[10,11], and adding grain refiner in the casting

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process of the melt is the most commonly used grain refinement method in Al alloy production[12]. Past research has shown that the mechanical properties of the Al-Si-Cu alloy have been improved obviously by the addition of 0.1 wt% of the master alloy, including the yield strength and elongation[13]. Rosenhain et al[14] found that adding extra Ti element before alloy casting can inhibit the growth of the columnar crystal region during solidification of the aluminum alloy. As an active element in the group IV_B, Ti is an effective grain refiner to improve the microstructure of aluminum alloy, thereby enhancing the mechanical properties[15]. Filippov et al showed that the highest tensile strength and hardness value was obtained for 0.5 wt% Ti specimen, and the second hardness value was Ti non-added specimen in as-cast condition. But the tensile strength decreased as the content of Ti increased because of the presence of cast defects and worse cast ability[16]. In addition, previous studies have found that the microstructure and mechanical properties of cast eutectic Al-Cu alloys are affected by different additions of Al-Ti-C master alloys (viz. 0, 0.2, 0.6, 1.0 and 2.0 wt%). On the one hand, the results show that the mean apparent lengths of coarse secondary phases were reduced to a minimum value when the master alloy addition reached 0.6 wt%[17]. One the other hand, the addition of minor Ti (0.04 wt%) to Al-Cu alloy can reduce the stress corrosion cracking resistance due to the reduction of the ability of inhibiting recrystallization during solid solution treatment[18]. Therefore, the Al-Cu alloy with a small amount of 0.2 wt% Ti was selected as the research object and the effects of conventional casting treatment, superheat treatment and thermal rate treatment on the solidification process, solidification structure and mechanical properties of Al-Cu-Ti alloys were investigated, which is expected to be beneficial for the development of new materials related to the solidification[19].

Furthermore, extensive results have demonstrated that the as-cast Al-Cu dendritic structure was eliminated by heat treatment resulting in improved mechanical properties[20,21]. Moreover, studies have shown that many alloy metastable non-uniformities are derived from the undesirable “defects” in the parent melt. A common explanation for this “defect” is that there are macroscopic and/or microscopic regions with different sizes, different solute enrichment levels and different structures in the melt, which are in a thermodynamic imbalance state[22,23]. However, the structure or state of the alloy melt after different melt heat treatments will change at a certain temperature, which is reflected in the jump change of the physical properties and temperature characteristics of the melt. It is worth noting that the “defects” inherited from the parent melt will decrease or even disappear after further thermal rate treatment[24]. Therefore, the heat treatment of alloy melts is of great significance for the solidification microstructure of alloys. When studying the effect of melt temperature treatment process on the structure and properties of A319 alloy[25], He et al melted the alloy in crucible at 750 °C which ensured that the raw materials could be melted and mixed evenly. Therefore, we chose 750 °C as the melting temperature. Moreover, Wang et al[26] found that the size of primary phase of binary Al alloy can be refined obviously and has long-term effect when the alloy is superheated to 900 °C. We expect that the optimal solidification structure can be achieved under the melt superheat treatment, so the temperature of 900 °C is set as the superheat treatment temperature. In the present study, the excellent structure of alloy melt can be obtained in the casting with the help of superheat and thermal rate treatment, thereby achieving the purpose of refining the grain size and improving the mechanical properties of alloy. Consequently, the heat treatment of alloy melts expands the traditional way of improving the mechanical properties of alloys through the solid heat treatment process and is conducive for the preparation of new materials with better mechanical properties.

1 Experiment

According to the liquidus temperature and eutectic reaction temperature of binary Al-Cu alloy, the melting temperature and superheat temperature of the alloy were selected as 750 and 900 °C, respectively. The proportioned Al ingot (99.98%), Cu block (99.998%) and Ti block (99.998%) placed in the high-purity alumina crucibles were superheated to 750 and 900 °C in a resistance furnace. Another set of samples were heat treated at 900 °C using cold chilling method, after which three sets of samples were poured into a sand mold. After cooling and solidification, three kinds of alloys, hypoeutectic $Al_{95}Cu_5$-0.2wt%Ti, eutectic $Al_{82.9}Cu_{17.1}$-0.2wt%Ti and hypereutectic $Al_{80}Cu_{20}$-0.2wt%Ti were obtained. The cast alloy was cut into a cylindrical specimen of 5 mm height and placed in a mounting press machine for inlaying. Finally, the mechanically polished samples were etched using Kohler reagent (95 mL H_2O+2.5 mL HNO_3+1.5 mL HCl+1.0 mL HF). The solidification morphology and phase distribution characteristics of Al-Cu-Ti alloys were observed on an M-4XC inverted metallographic microscope.

The constituent phases of Al-Cu-Ti alloys were analyzed by X-ray diffraction (XRD, Bruker D8 Advance). The microscopic morphology and phase composition of alloys were examined under a scanning electron microscope (SEM, JEOL, JXA-8100, Tokyo, Japan) equipped with an energy-dispersive spectrum. In order to characterize the phase transition of alloys, the alloy samples were tested in a differential scanning calorimeter (DSC, Mettler-Toledo, Zurich, Switzerland) in the temperature range of 25~1000 °C. The heating and cooling rates were set as 10 °C/min. The hardness of alloys was measured using a Brinell hardness testers of HBRVU-187.5 under the loading pressure of 613 N and loading time of 30 s. The uniaxial tension test was performed at room temperature with a tensile rate of 0.5 mm/min on a DWG-1060 microcomputer universal testing machine. And a computer with data acquisition software was used to collect the data.

2 Results and Discussion

2.1 Phase constitution and solidification microstructures

The XRD patterns of samples, including $Al_{95}Cu_5$-0.2wt%Ti alloy, $Al_{82.9}Cu_{17.1}$-0.2wt%Ti alloy, and $Al_{80}Cu_{20}$-0.2wt%Ti alloy, were obtained and the Miller indices were indexed in Fig.1. The main crystalline phases of the alloy are Al (Joint Committee on Powder Diffraction Standard cards, No. 04-0487) and Al_2Cu (No. 25-0012). The Al presents a simple cubic structure with the space group of Fm-3m (a=b=c=0.405 nm) while the Al_2Cu exhibits a tetragonal structure with the space group of I4/mcm (a=b=0.607 nm, c=0.487 nm). The phases related to Ti element were not observed due to the relatively small amounts in all the samples. As seen in Fig.1, the intensity of (200) peak for Al phase weakens due to the increasing of Cu. While the (310) peak for Al_2Cu strengthens with the Cu increasing, indicating the phase transition from cubic to tetragonal[27-30]. Fig.2 is the SEM image and EDS spectra of $Al_{95}Cu_5$ alloy with the addition of 0.2 wt% Ti. It can be seen that the black region is Al phase and the white portion is Al_2Cu phase. Moreover, the eutectic microstructure distributes in the form of lamellar on the Al matrix.

Fig.3 shows the solidification microstructure of $Al_{95}Cu_5$ hypoeutectic alloy with the addition of 0.2wt%Ti after different melt heat treatment processes. Combined with the Al-Cu binary phase diagram, it is concluded that the microstructure of three kinds of Al-Cu alloys is mainly composed of the primary α-Al phase directly precipitated from the liquid phase and the eutectic structure (α-Al+θ-Al_2Cu) from the XRD patterns. The morphology of primary α-Al phase is approximately circular, which is a typical characteristic of non-facet solidification[31], and the eutectic structure is distributed in the form of layer at the grain boundaries. It is found in Fig.3 that the primary α-Al phase is large after conventional casting. However, the primary α-Al phase of $Al_{95}Cu_5$ hypoeutectic alloy has a tendency to decrease after superheat treatment. Furthermore, the average grain size in the whole field of view under different melt heat treatment processes in Fig.3 was calculated by analysis software, which are 140.0, 95.9 and 80.3 μm, respectively. It can be concluded that the average

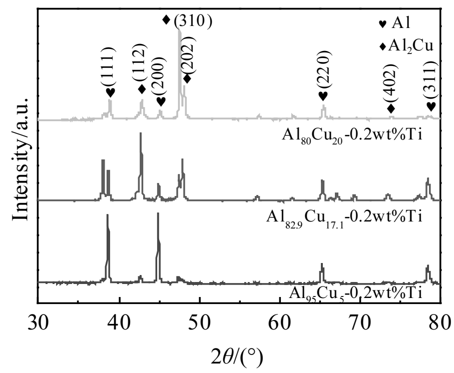

Fig.1 XRD patterns of Al-Cu-Ti alloys after conventional casting

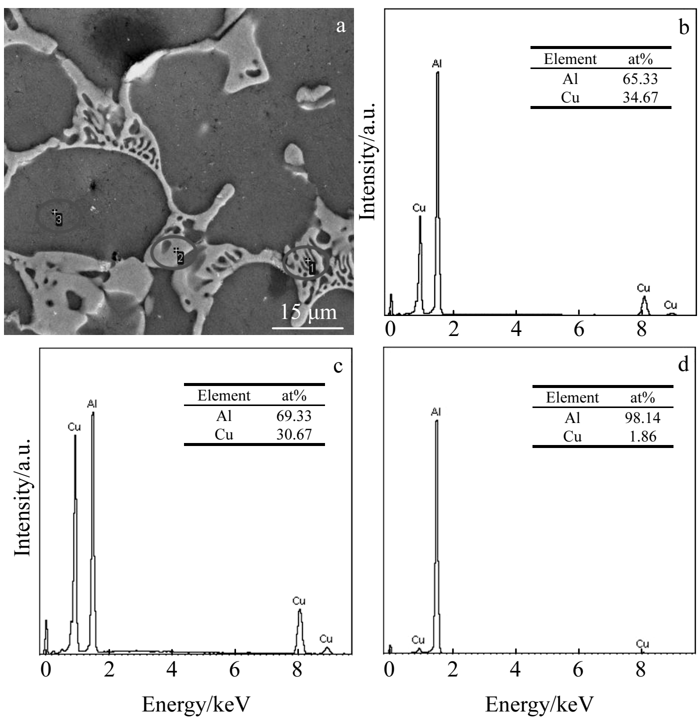

Fig.2 SEM image (a) and EDS spectra of $Al_{95}Cu_5$-0.2wt%Ti alloy cast into sand mold after conventional casting: (b) point 1, (c) point 2, and (d) point 3

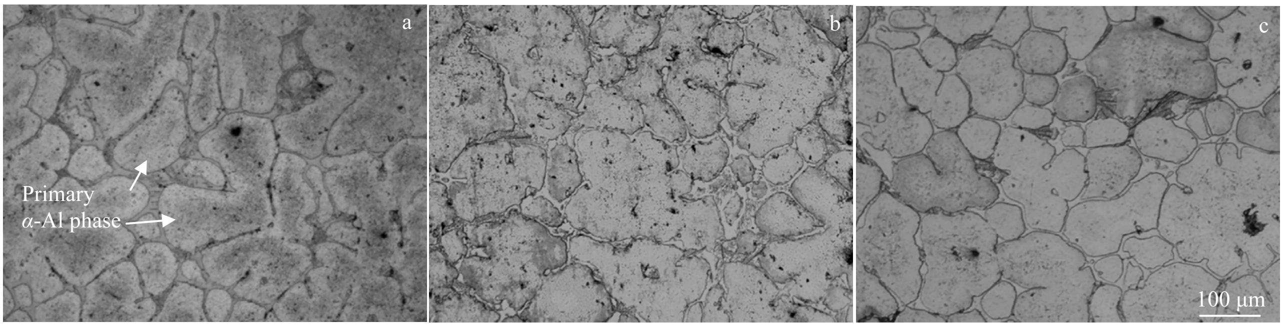

Fig.3 Solidification microstructures of $Al_{95}Cu_5$-0.2wt%Ti alloy after different melt heat treatment processes: (a) conventional casting, (b) superheat treatment, and (c) thermal rate treatment

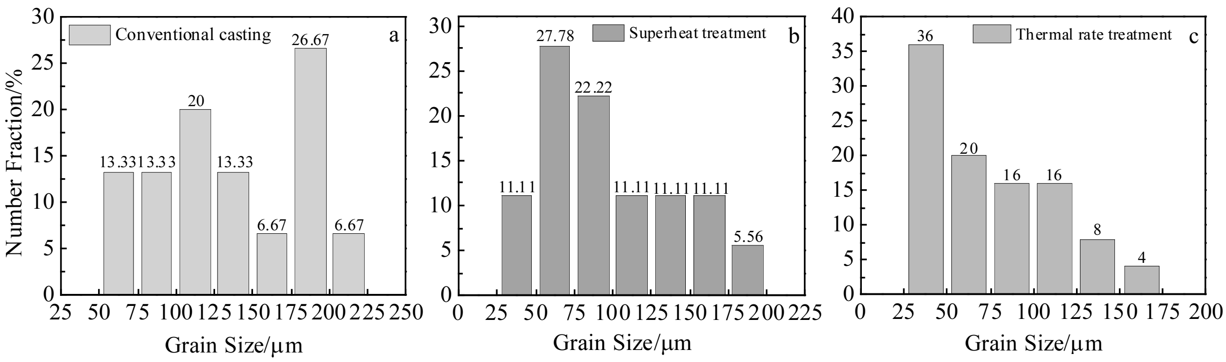

Fig.4 2D size distribution of the grains in the $Al_{95}Cu_5$-0.2wt%Ti alloy after different melt heat treatment processes in Fig.3: (a) conventional casting, (b) superheat treatment, and (c) thermal rate treatment

grain size of the $Al_{95}Cu_5$-0.2wt%Ti alloy is significantly reduced after the superheat treatment and thermal rate treatment. From Fig.4a and 4b, it can be ascertained that the grain sizes under conventional casting and superheat treatment are quite different, and there are many small grain sizes under superheat treatment. In addition, it is found from Fig.4c that the distribution range of grain size decreases (the solidified structure is obviously more uniform) under thermal rate treatment, and most of the grain size is concentrated in the range of 25~50 μm.

Fig.5 shows the microstructure of the $Al_{82.9}Cu_{17.1}$ eutectic alloy with the addition of 0.2wt%Ti after different melt heat treatment processes. It can be seen that the solidification structure of $Al_{82.9}Cu_{17.1}$-0.2wt%Ti eutectic alloy only contains eutectic structure (α-Al+θ-Al_2Cu). The eutectic microstructure is lamellar or mesh-like with a part of coarse grains interposed. However, the interlaminar spacing of eutectic microstructure of $Al_{82.9}Cu_{17.1}$-0.2wt%Ti alloy after superheat treatment decreases. After thermal rate treatment, the microstructure of $Al_{82.9}Cu_{17.1}$-0.2wt%Ti is refined evidently and the average spacing of eutectic microstructure is the smallest, just as shown in Fig.5c.

Fig.6 is the solidification morphology of $Al_{80}Cu_{20}$ hypereutectic alloy with the addition of 0.2wt%Ti after different melt heat treatment processes. The microstructure of $Al_{80}Cu_{20}$-0.2wt%Ti alloy after conventional casting consists of primary θ-Al_2Cu phase in the shape of block and eutectic structure (α-Al+θ-Al_2Cu). The reason is that the θ-Al_2Cu phase in hypereutectic alloys exhibit faceted growth properties, and most of them have obvious corners[32]. Similar to the $Al_{82.9}Cu_{17.1}$-0.2wt%Ti eutectic alloy, the interlamellar spacing of eutectic structure of the $Al_{80}Cu_{20}$-0.2wt%Ti hypereutectic alloy after superheat treatment is reduced. After thermal rate treatment, Fig.6c shows that the primary θ-Al_2Cu phase distributes uniformly and the grain size is significantly reduced. It is concluded that the refinement effect of thermal rate treatment of alloy melt in the three processes above is most remarkable. During thermal rate treatment, the alloy melt is rapidly cooled to the casting temperature by the raw aluminum ingot. Under rapid mechanical agitation, the raw ingot promptly absorbs heat and melts so that the melt is cooled and the melt obtains a large cooling rate. At this time, the atomic nucleation and growth time in the melt are greatly shortened. An excellent melt structure at high temperatures under such non-equilibrium conditions can be obtained and retained, resulting in a significant refinement of microstructure and a reduction in the

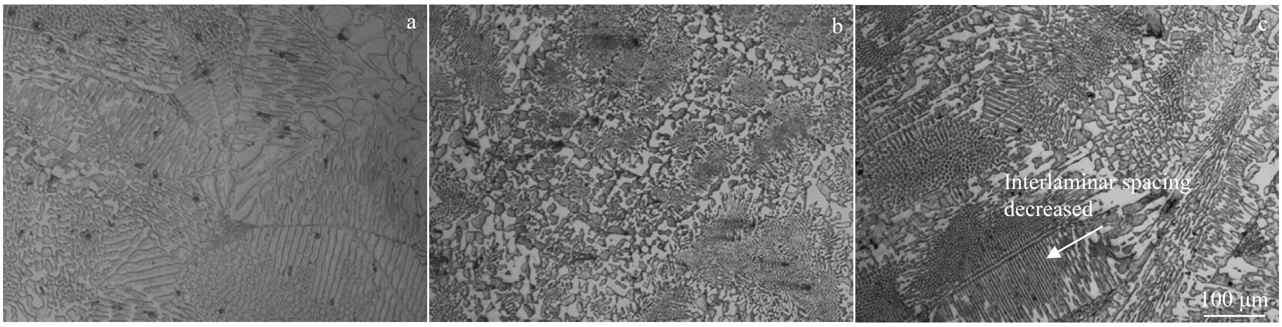

Fig.5 Solidification microstructures of $Al_{82.9}Cu_{17.1}$-0.2wt%Ti after different melt heat treatment processes: (a) conventional casting, (b) superheat treatment, and (c) thermal rate treatment

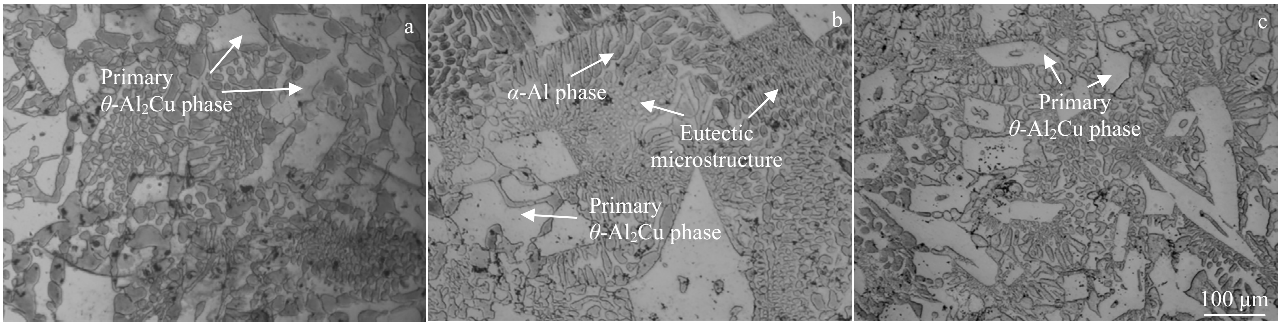

Fig.6 Solidification microstructures of $Al_{80}Cu_{20}$-0.2wt%Ti alloy after different melt heat treatment processes: (a) conventional casting, (b) superheat treatment, and (c) thermal rate treatment

average spacing of eutectic structure of alloy[33,34].

2.2 Phase transition behavior

It is well known that DSC test can measure many thermodynamic and kinetic parameters[35], such as specific heat, reaction heat, and transition heat. And DSC curve directly reflects the heat energy change of the sample during thermal reaction; therefore we can study the thermal reaction of the sample at constant heating rate accordingly. In the present work, the phase transition behavior of Al-Cu-Ti alloys was studied by DSC curves obtained at a rate of 10 °C/min. Fig.7a shows the DSC curves of the Al-Cu-Ti alloy after the conventional casting. From the binary phase diagram of Al-Cu alloy, it can be seen that the $Al_{95}Cu_5$ hypoeutectic alloy first precipitates a primary α-Al phase, and then the eutectic reaction occurs with the decrease of melt temperature. For the eutectic composition of $Al_{82.9}Cu_{17.1}$, only one eutectic reaction takes place during the melt cooling process. So, it can be seen that the $Al_{95}Cu_5$ hypoeutectic alloy with the addition of 0.2 wt% Ti has two endothermic peaks; the endothermic peak at 544 °C corresponds to the melting of eutectic composition (the eutectic reaction temperature of Al-Cu alloy is 548.2 °C), while the other peak at 633 °C represents the melting of α-Al phase. Similarly, the endothermic peak of $Al_{82.9}Cu_{17.1}$-0.2wt%Ti alloy also corresponds to the melting of eutectic composition. It should be pointed out that the $Al_{80}Cu_{20}$-0.2wt%Ti hypereutectic alloy has only one endothermic peak. On the one hand, it may be not detected during the test process due to the small quantity of precipitated θ-Al_2Cu. The temperature of precipitation of θ-Al_2Cu phase is close to that of eutectic reaction; therefore it may coincide with the endothermic peak representing eutectic reaction. Fig.7b is the DSC curve of $Al_{80}Cu_{20}$-0.2wt%Ti alloy after different melt heat treatments. According to Speyer's formula, the latent heat of melting (crystallization) is proportional to the peak area[36-38]. It can be seen that the peak area in the DSC curve of $Al_{80}Cu_{20}$-0.2wt%Ti alloy after conventional casting is the largest, and thus the corresponding latent heat of fusion is largest. The $Al_{80}Cu_{20}$-0.2wt%Ti alloy after the thermal rate treatment has the smallest area of endothermic peak and the lowest latent heat of fusion. Sun et al[39] found that the reason for the reduction in the latent of fusion may be mainly considered as the degradation of the chemical structure of Al alloys in DSC tests. As shown in Fig.6, the solidification structures obtained under different melt heat treatment processes are quite different, which will be reflected in the difference of latent heat of melting. In addition, the thermal rate treatment method in the present paper is cold material chilling method. As is known to all, the newly added cold

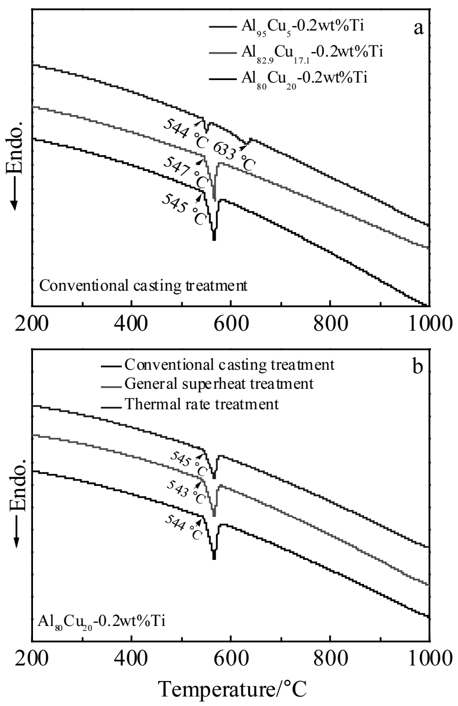

Fig.7 DSC heating curves of Al-Cu-Ti alloy cast into the sand mold after conventional casting (a) and $Al_{80}Cu_{20}$-0.2wt%Ti after different heat treatment processes (b)

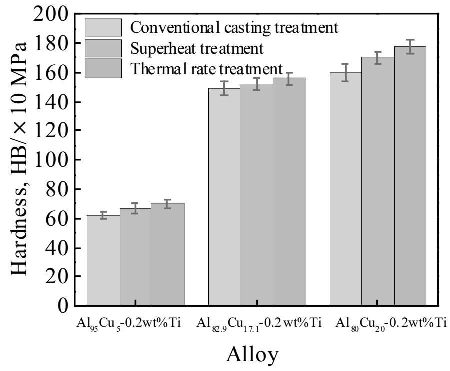

Fig.8 Hardness of Al-Cu-Ti alloys after different heat treatment processes

material will destroy the equilibrium formed in the melt and change its structure. Due to the rapid melting of cold material in high temperature melt and the effect of mechanical stirring, it will lead to relatively large energy/composition fluctuations in the melt simultaneously. The melting latent heat of the alloy decreases with the higher energy state and less heat required for melting[40]. Moreover, as is well known that the finer the grain size of alloy, the higher the total interfacial energy in the corresponding volume, which indicates that the lower latent heat of fusion is needed to achieve melting. After the solidification of alloy melt, the grain size of alloy is refined significantly, thereby releasing less latent heat of crystallization[41].

2.3 Mechanical properties

It is generally known that the true tensile strength testing of solidified alloys gave inconsistent results with a wide scatter due to their strong dependence on solidified sample surface quality. Therefore, the mechanical properties were monitored by hardness testing, which is one of the easiest and most straightforward techniques. Fig.8 is the Brinell hardness of Al-Cu-Ti alloys after different melt heat treatment processes. It can be seen that the Al-Cu alloy with the addition of 0.2 wt% Ti after thermal rate treatment has the highest hardness and the hardness of alloy after conventional casting is the smallest. Due to the thermal rate treatment, the melt structure of alloy is changed or improved. As the cooling rate is relatively faster under the non-equilibrium solidification conditions, the atom clusters in the melt have solidified before they are rapidly aggregated which effectively increases the nucleation core in the alloy melt during solidification. In addition, the alloy melt structure becomes uniform and dense at the higher temperature, so the grain size or the microstructure which has less defects of the alloy is effectively refined and the phase distribution of microstructure is more uniform[42]. When the microstructure of samples is refined, the number of corresponding grain boundaries increases. As the interface between grains with different orientations, grain boundaries can hinder dislocation movement and improve material strength. Generally speaking, the mechanical properties (strength, plasticity and toughness) of fine-grained metals are always better than those of coarse-grained metals. In addition, it should be pointed out that the increase in hardness after melt heat treatment can be attributed to the supersaturated solid solution of α-Al phase[43]. On the one hand, the hardness of θ-Al_2Cu is relatively larger than that of α-Al, so the overall hardness increases with the content of Cu increasing. On the other hand, however, the θ-Al_2Cu phase precipitated from the samples with the same composition during melt cooling is certain. Therefore, it is difficult to see significant differences in the hardness, especially for $Al_{95}Cu_5$-0.2wt%Ti and $Al_{82.9}Cu_{17.1}$-0.2wt%Ti alloy.

Tensile tests were carried out on three kinds of samples with different components after different melt heat treatments, just as shown in Fig.9. It can be found that the tensile strength of alloy increases with the increase of Cu content. It is noteworthy that the trend is consistent with the hardness value. Before reaching the eutectic point, the eutectic structure increases with the increase of Cu content, and then the primary θ-Al_2Cu phase gradually precipitates with the further increase of Cu content. As an intermetallic compound, the θ-Al_2Cu phase can play the role of dispersion strengthening. It can be found from the solidification structure of Al-Cu-Ti alloy that the distribution of θ-Al_2Cu is more uniform with

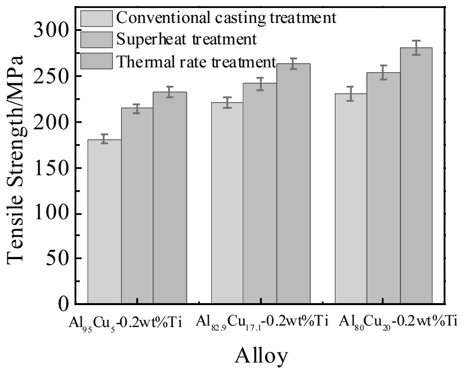

Fig.9 Tensile strength of Al-Cu-Ti alloys after different heat treatment processes

superheat treatment and thermal rate treatment. Therefore, the strength of materials can be enhanced. After superheat treatment, the segregation of elements between dendrites is weakened. In addition, the dendrite spacing is reduced, and the number of strengthening phases is increased, suggesting the size and distribution of strengthening phase are improved, and as a result, the mechanical properties of alloys are improved[44]. Moreover, the supercooling degree of melt is increased by thermal rate treatment, which leads to further refinement of grains in the alloy. Therefore, it is shown that the tensile properties of the alloys after superheat treatment are higher than those after conventional casting treatment, while the tensile properties of the samples after thermal rate treatment are the best.

3 Conclusions

1) The thermal rate treatment process has the best grain refinement effect and the mechanical properties of Al-Cu-Ti alloy after thermal rate treatment are the best, which demonstrates that thermal rate treatment process could improve the "defects" in the alloy melt.

2) The thermal rate treatment could lead to relatively large energy/composition fluctuations, which effectively increases the nucleation rate resulting in a better refinement effect and improves the mechanical properties of alloy.

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不同熔体热处理工艺后 **Al-Cu-Ti** 合金的晶粒细化及力学性能改善

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摘 要：研究了添加细化剂和不同热处理工艺（过热处理和热速处理）对 Al-Cu 合金的改性。分析了 Al-Cu-Ti 合金的相组成、凝固微观组织和力学性能。此外，采用差示扫描量热法（DSC）研究了 Al-Cu-Ti 合金在不同熔体热处理工艺后的熔体结构转变行为。实验结果表明，热速处理极大地细化了 Al-Cu-Ti 合金晶粒，且力学性能得到有效改善。通过对热力学相变分析，发现 Al-Cu-Ti 合金经过热处理和热速处理后的熔化潜热随着界面能的增大而变小，从而在一定程度上细化了合金的微观组织。

关键词：熔体热处理工艺；熔体结构；凝固组织；晶粒细化；力学性能

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