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ARTICLE

Effect of Al-5Ti-0.8C Master Alloy on Microstructure and Elevated Temperature Mechanical Properties of Al-Cu-Mn Alloy

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Abstract: A new kind of Al-5Ti-0.8C master alloy was fabricated using a self-propagating combustion reaction method and dilution treatment, and the effects of different Al-5Ti-0.8C master alloy contents (0wt%, 0.1wt%, 0.3wt%, 0.5wt%) on the microstructure and elevated temperature mechanical properties of Al-Cu-Mn alloy were investigated. The results show that the Al-5Ti-0.8C master alloy refines the Al-Cu-Mn alloy, increases the amount of $\theta'(Al_2Cu)$ precipitates, and reduces their size during heat treatment, resulting in the grain refinement. Moreover, this new master alloy obviously improves the elevated temperature mechanical properties of Al-Cu-Mn alloy, mainly due to the precipitation strengthening caused by the uniform distribution of second phases and fine $\theta'(Al_2Cu)$ precipitates, and the formation of $Al_3(Ti, Zr)$ nanoparticles with high thermal stability. Furthermore, the microstructure and mechanical properties of Al-Cu-Mn alloy with 0.3wt% Al-5Ti-0.8C master alloy show the optimal status.

Key words: Al-5Ti-0.8C master alloy; Al-Cu-Mn alloy; grain refinement; precipitation strengthening; elevated temperature mechanical properties

Al-Cu-Mn alloy has been the material of research focus in the fields of automobile manufacturing and aircraft construction as a structural lightweight metal material, owing to its excellent mechanical properties at room temperature[1-3]. However, its worldwide applications are restricted because of the elevated temperature properties. For example, the work temperatures of diesel engine cylinder heads are generally in the range of 473~573 K[4,5], but the elevated temperature properties of casting Al-Cu-Mn alloy degenerate due to the softening and coarsening of θ' phase[3,6,7]. Moreover, Al-Cu-Mn alloy is prone to hot tearing[8], resulting in a quick decrease in the mechanical properties. Recently, many experiments have been conducted to improve the elevated temperature properties of casting Al-Cu-Mn alloy and avoid hot tearing. Grain refinement is an effective method[9-11], and master alloys are confirmed as the most economical grain refiner[12-14].

Al-Ti-C master alloy containing TiC particles is one of the effective grain refiners for Al-Cu alloy, and its effects on the microstructure and mechanical properties of Al-Cu alloys have been widely investigated. Ding et al[15] found that Al-24Ti-6C master alloy has an excellent grain refining effect on Al-4.5Cu alloy, and TiC particles are stable in Al alloy, which is consistent with the results of Al-5Ti-0.75C refined Al-5Cu alloy[16]. Furthermore, Wang et al[17] reported that Al-5Ti-0.4C master alloy refines the Al-5Cu alloy, but $Ti(Al, Cu)_2$ forms due to the interaction of Al_3Ti and Al_2Cu, resulting in a reduced grain refining effect. Huang et al[18] studied the effect of Al-5Ti-0.2C master alloy on the mechanical properties of Al-Zn-Cu-Mg alloy, and found that the strength and hardness of the alloy improve due to the uniform distribution of secondary phases. However, these studies focused mainly on the microstructure evolution of Al-Cu alloys refined by different master alloys. Few researches reported the optimal amount of Al-Ti-C master alloy and its effect on the elevated temperature mechanical properties of Al-Cu-Mn alloy.

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In the present study, a new kind of Al-5Ti-0.8C master alloy was prepared using a self-propagating combustion reaction method, and its effects on the microstructure and elevated temperature mechanical properties of Al-Cu-Mn alloy were studied. Based on the experimental results, the optimal amount of the new Al-Ti-C master alloy was determined, and the mechanism of its effect on the mechanical properties of Al-Cu-Mn alloy at high temperature was identified.

1 Experiment

Ti powder (99.9wt%), Al powder (99.9wt%) and graphite powder (99.9wt%) were used as feedstock. Firstly, the feedstock was mixed uniformly with 50wt% Al powder and 50wt% Ti/C powder, in which the ratio of Ti powder to graphite powder is 6:1. According to Yang et al[19], when Ti/C ratio is high, the self-propagation high-temperature synthesis (SHS) reaction process has no extra graphite source to form TiC particles. On the contrary, when Ti/C ratio is low, the Al_4C_3 hard brittle phase forms by extra graphite source. Thus, Ti:C=6:1 was selected as the optimal ratio. Secondly, the feedstock was pressed into block and the blocky material was heated to 950 °C using a 1000 W laser under Ar protection gas atmosphere and then ignited. After the self-propagating combustion reaction finished, the product Al-43Ti-7C was air-cooled. Thirdly, the reaction product was diluted with pure aluminum according to the proportion of Ti element to obtain the Al-5Ti-0.8C master alloy.

Al-Cu-Mn alloy (5.0wt% Cu, 0.5wt% Mn, 0.2wt% Ti, 0.15wt% V, 0.1wt% Cd, 0.15wt% Zr, 0.002wt% B) was prepared in an electrical resistance furnace at 730 °C. 0wt%, 0.1wt%, 0.3wt% and 0.5wt% Al-5Ti-0.8C master alloy was added separately into the Al-Cu-Mn alloy, and the melt was heated to 750 °C. Dry C_2Cl_6 tablets were used to degas the container, and the amount of C_2Cl_6 tablets was equal to that of 0.6wt% melt. After stirring for 1 min, the melt was cast in an iron mould at 710 °C, which was firstly preheated to 200 °C. The casting alloys were homogenized at 538 °C for 14 h, quenched at 50 °C in hot water, and then aged at 155 °C for 8 h.

Samples were cut from the centre of the Al-5Ti-0.8C master alloy and Al-Cu-Mn alloys, mechanically ground, polished and etched with Keller's reagent. A high scope video microscope (HSVM) and a scanning electron microscope (SEM) equipped with energy disperse spectroscopy (EDS) were used for microstructure observation and elemental analysis. X-ray diffraction (XRD) was used to examine phase composition. The microstructure and phase distribution of Al-Cu-Mn alloy after heat treatment were examined using transmission electron microscope (TEM). The standard thin foils for TEM observation were manually ground, polished, and milled using an ion polishing system with a working voltage of 5 kV. The grain size of the alloy was measured based on the linear intercept method with ImageJ software, and the data were taken from at least 30 regions for each sample.

Dog-bone-shaped tensile specimens with a gauge length of 40 mm and a cross-section diameter of 8 mm were sectioned from the Al-Cu-Mn alloy after heat treatment, and tensile tests were carried out at room temperature and elevated temperatures.

2 Results and Discussion

2.1 Microstructure of Al-5Ti-0.8C master alloy

Fig. 1 shows the XRD result of Al-5Ti-0.8C master alloy and suggests that the new master alloy mainly consists of α-Al, Al_3Ti, and TiC phases. The microstructures of Al-5Ti-0.8C master alloy, as shown in Fig. 2, indicate that Al_3Ti and TiC particles are uniformly dispersed in Al matrix. Based on the result of EDS analysis, the fine particles are TiC, and the plate-like particles are Al_3Ti. Moreover, the sizes of the dispersedly distributed TiC particles are in the range of 0.2~1.0 μm (Fig. 2b). According to the previous studies[20-22], the grain refinement effect improves with the increase in the dispersion of particles. Thus, the Al-5Ti-0.8C master alloy prepared through the self-propagating combustion reaction method is likely to exhibit an excellent grain refinement effect.

2.2 Effect of Al-5Ti-0.8C master alloy content on microstructure of Al-Cu-Mn alloy

The microstructures of casting Al-Cu-Mn alloys with different Al-5Ti-0.8C master alloy contents are shown in Fig. 3. Fig. 3a indicates that the unrefined Al-Cu-Mn alloy consists of equiaxed dendrites with an average grain size of 100 μm. After the addition of 0.1wt% and 0.3wt% Al-5Ti-0.8C master alloy, the average grain sizes of the Al-Cu-Mn alloy decrease to 80 and 50 μm, respectively (Fig.3b and 3c). Grain coarsening occurs when the master alloy content increases to 0.5wt% (Fig.3d), and the average grain size is 60 μm. It can be concluded that with increasing the content of Al-5Ti-0.8C master alloy, the grain size of Al-Cu-Mn alloy firstly decreases and then increases. The wetting angle of TiC particles is relatively large due to its convex structure, and the fine TiC particles effectively promote the heterogeneous nucleation, resulting in the decrease of grain size. As the Al-5Ti-0.8C master alloy content increases, the agglomeration phenomenon becomes more obvious and TiC particles form large clusters. Thus, the refining effect weakens and the grain size increases.

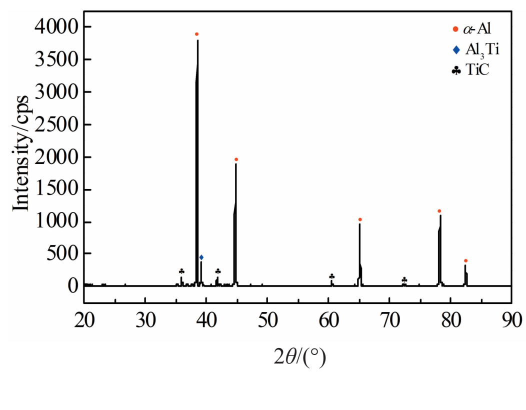

Fig.1 XRD pattern of Al-5Ti-0.8C master alloy

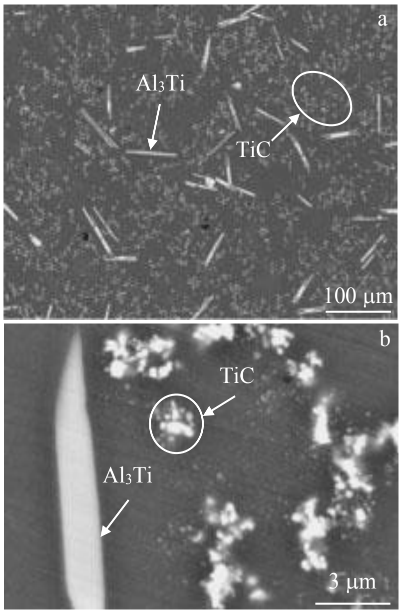

Fig.2 SEM images of Al-5Ti-0.8C master alloy at low (a) and high (b) magnification

Fig. 4 shows the SEM images of Al-Cu-Mn alloy with different Al-5Ti-0.8C master alloy contents. The reticular θ (Al_2Cu) phases in the unrefined Al-Cu-Mn alloy are precipitated along the grain boundaries (Fig.4a). It can be seen that θ(Al_2Cu) phases become discontinuous and thin when 0.1wt% Al-5Ti-0.8C master alloy is added (Fig. 4b). After the addition of 0.3wt% Al-5Ti-0.8C master alloy, θ(Al_2Cu) phases transform to stripe and block shape (Fig. 4c), and the amount of θ(Al_2Cu) phases also increases with the increase of the master alloy content, as shown in Fig.4d.

2.3 Effect of heat treatment on microstructure of refined Al-Cu-Mn alloy

Previous studies[23-25] indicated that the improvement of mechanical properties of Al-Cu alloy refined by master alloys results from the formation of θ' (Al_2Cu) precipitates during heat treatment. Thus, the effect of Al-5Ti-0.8C master alloy on θ'(Al_2Cu) phases after heat treatment was studied. Fig.5 shows the TEM images of Al-Cu-Mn alloy refined by different contents of Al-5Ti-0.8C master alloy. As indicated in Fig. 5a, only a few layered θ' (Al_2Cu) precipitates occur with an average length of 80 nm in the unrefined Al-Cu-Mn alloy. After the addition of Al-5Ti-0.8C master alloy, the needle-like θ' (Al_2Cu) phases are precipitated and regularly distributed. The average length of θ' (Al_2Cu) precipitates decreases to 60, 30, and 50 nm in the Al-Cu-Mn alloys refined by 0.1wt%, 0.3wt%, and 0.5wt% Al-5Ti-0.8C master alloy, respectively, and the amount of θ' (Al_2Cu) precipitates increases with the increase of Al-5Ti-0.8C master alloy contents, which suggests that Al-5Ti-0.8C master alloy facilitates the formation of θ' (Al_2Cu) precipitates and reduces their sizes. Fig. 6 presents the size distribution of θ' (Al_2Cu) precipitates in Al-Cu-Mn alloys with 0wt% and 0.3wt% Al-5Ti-0.8C master alloy.

According to previous studies[26,27], the excess vacancies in Al-Cu alloy play an important role in the formation of θ'(Al_2Cu) precipitates, indicating that the density of vacancies determines the amount of θ' (Al_2Cu) precipitates. On the one hand, the vacancies in the matrix at the solution-treatment temperature migrate to grain boundaries during quenching process, suggesting that the grain size affects the precipitation

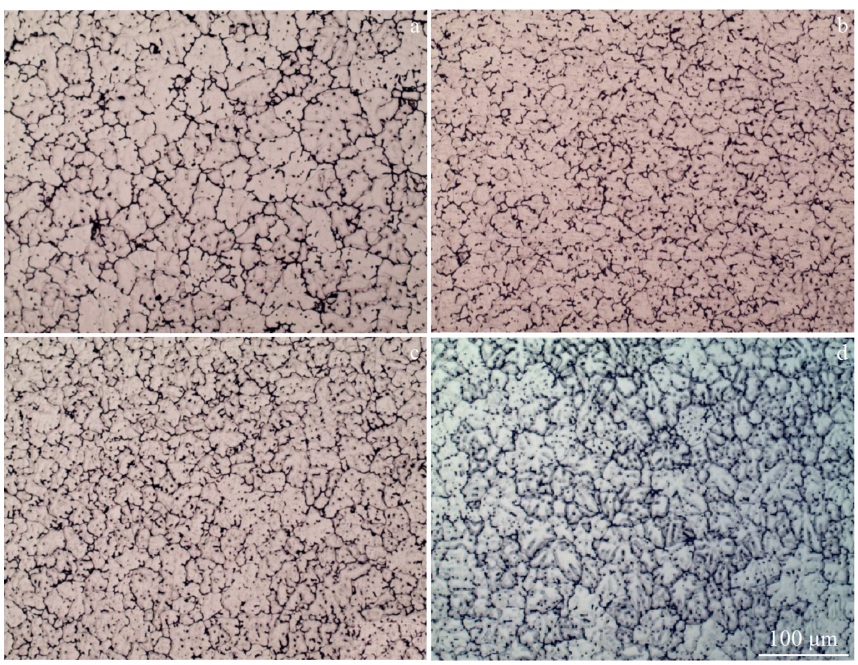

Fig.3 Microstructures of Al-Cu-Mn alloy with different Al-5Ti-0.8C master alloy contents: (a) 0wt%, (b) 0.1wt%, (c) 0.3wt%, and (d) 0.5wt%

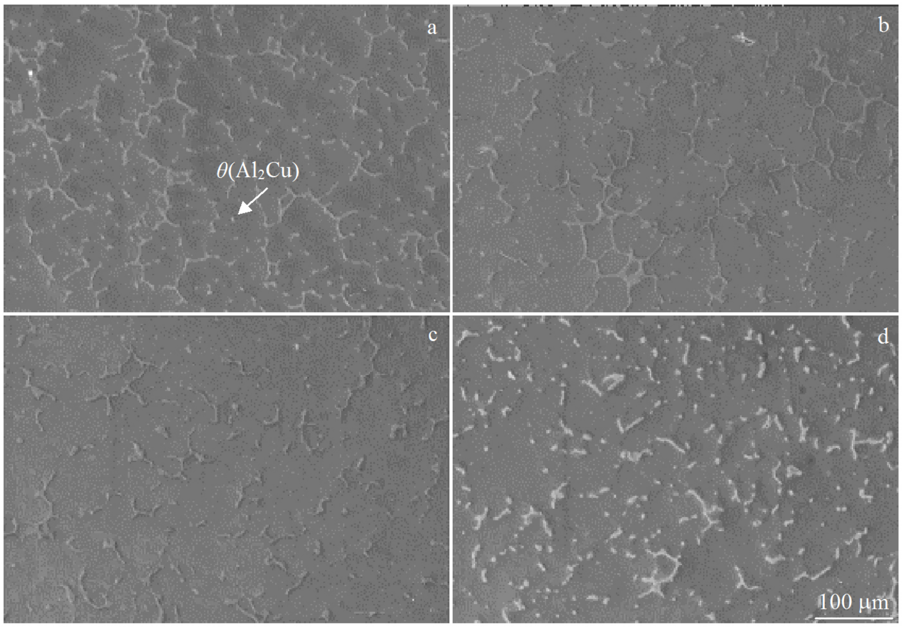

Fig.4 SEM images of Al-Cu-Mn alloy with different Al-5Ti-0.8C master alloy contents: (a) 0wt%, (b) 0.1wt%, (c) 0.3wt%, and (d) 0.5wt%

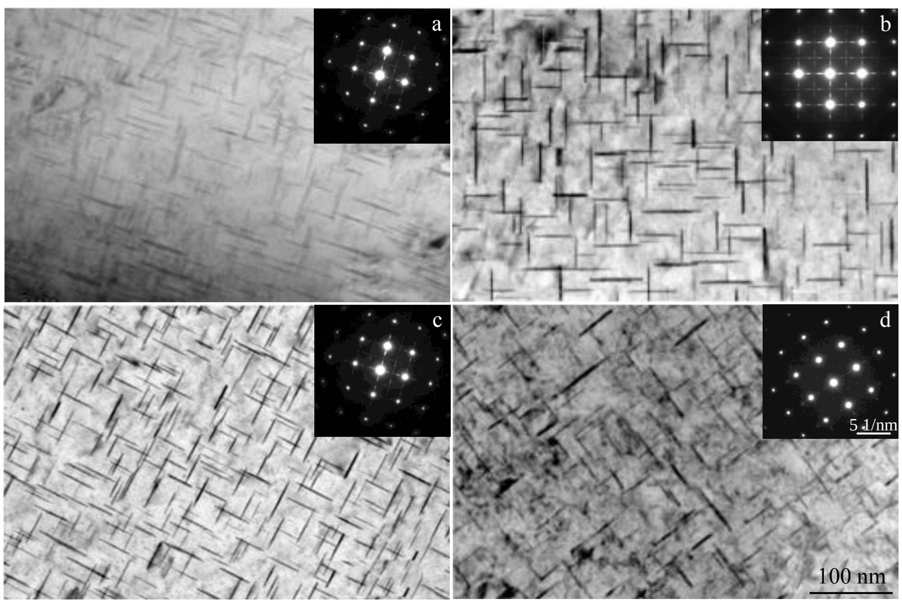

Fig.5 TEM images of Al-Cu-Mn alloy refined by different contents of Al-5Ti-0.8C master alloy after heat treatment: (a) 0wt%, (b) 0.1wt%, (c) 0.3wt%, and (d) 0.5wt%

kinetics, and the precipitate nucleation occurs preferentially at grain boundaries[28]. As shown in Fig. 3, the Al-5Ti-0.8C master alloy has an excellent grain refinement effect on the Al-Cu-Mn alloy with the average grain size of Al-Cu-Mn alloy decreasing after Al-5Ti-0.8C master alloy addition. Thus, the amount of grain boundaries increases markedly, providing more places for $\theta'(Al_2Cu)$ precipitate nucleation. According to the previous research, on the other hand, plenty of vacancies are generated when Cu atoms dissolve into the Al matrix during the solution treatment, which is beneficial to the formation of $\theta'(Al_2Cu)$ precipitates[29]. Fig.7 shows the microstructures of the Al-Cu-Mn alloys refined by Al-5Ti-0.8C master alloy after solution treatment. Compared with the unrefined alloy, less $\theta(Al_2Cu)$ phases are distributed along the grain boundary in the refined Al-Cu-Mn alloy, suggesting that most of the Cu atoms dissolve into the matrix during heat

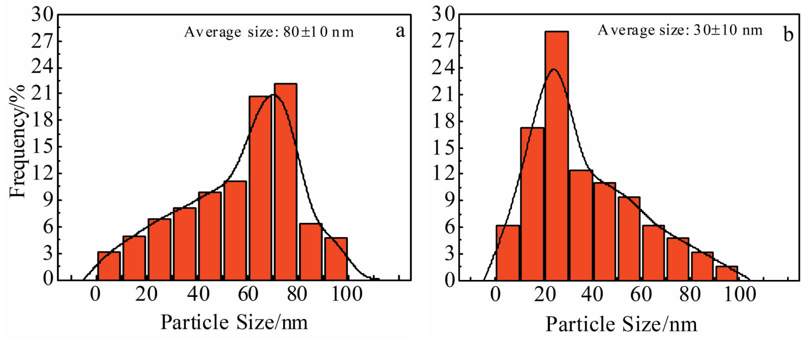

Fig.6 Particle size distribution of $\theta'(Al_2Cu)$ participates in Al-Cu-Mn alloys with 0wt% (a) and 0.3wt% (b) Al-5Ti-0.8C master alloy

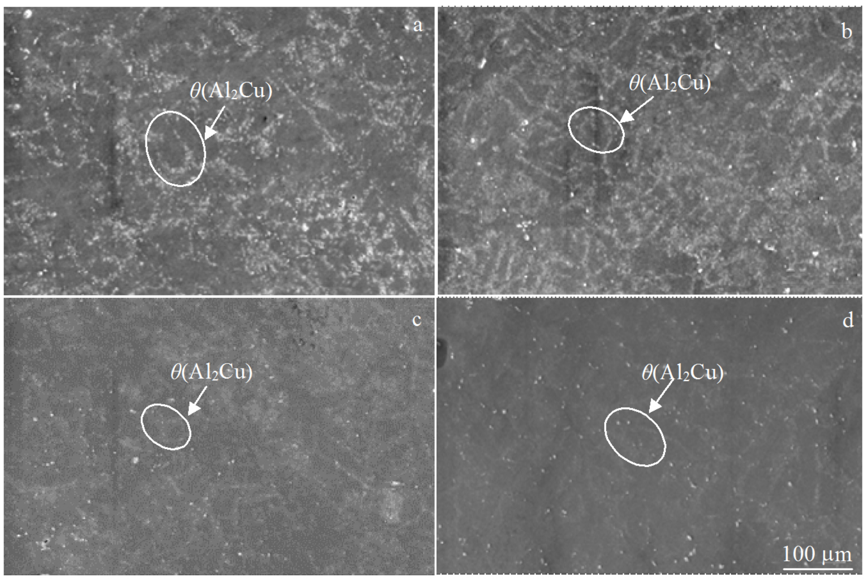

Fig.7 Microstructures of Al-Cu-Mn alloy refined by different contents of Al-5Ti-0.8C master alloy after heat treatment: (a) 0wt%, (b) 0.1wt%, (c) 0.3wt%, and (d) 0.5wt%

treatment. Moreover, the amount of $\theta(Al_2Cu)$ phases firstly decreases and then increases with increasing the content of Al-5Ti-0.8C master alloy. Due to the large number of $\theta'(Al_2Cu)$ precipitates formed simultaneously, there are not enough solute atoms, which inhibits the growth of $\theta'(Al_2Cu)$ precipitates and results in the small size.

The blocky phase in the refined alloy with 0.3wt% Al-5Ti-0.8C master alloy was analysed by TEM, as shown in Fig.8. It can be confirmed that the blocky phase is $Al_3(Zr, Ti)$ phase.

2.4 Mechanical properties of Al-Cu-Mn alloy

Fig.9 shows the mechanical properties of Al-Cu-Mn alloy refined by different contents of Al-5Ti-0.8C master alloy after heat treatment. It is indicated that the ultimate tensile strength (UTS), yield strength (YS), and elongation (EL) of the tested alloys obviously improve. Moreover, the mechanical properties of Al-Cu-Mn alloy firstly improve and then degenerate with the increase of Al-5Ti-0.8C master alloy content, and the alloy obtains the optimal properties when it contains 0.3wt% Al-5Ti-0.8C master alloy. For Al-Cu-Mn alloy refined by 0.3wt% Al-5Ti-0.8C master alloy, the UTS, YS, and EL is 499 MPa, 435 MPa, and 18.8%, increased by 8.2%, 10.1%, and 91.8% compared with those unrefined alloy, respectively. The fractographies of Al-Cu-Mn alloy are shown in Fig.10. It can be seen that the unrefined Al-Cu-Mn alloy shows a brittle fracture nature, while the Al-Cu-Mn alloy refined by 0.3wt% Al-5Ti-0.8C master alloy exhibits large areas of tear ridges and dimples, indicating that the refined alloy has excellent plasticity.

The microstructure and mechanical properties of Al-Cu-Mn alloy with 0.3wt% Al-5Ti-0.8C master alloy are found to be optimal. Therefore, the mechanical properties of the 0.3wt% Al-5Ti-0.8C refined alloy were tested at elevated temperatures, and the results are indicated in Fig.11. It can be seen that the tensile strength of all the tested alloys decreases significantly at elevated temperatures. Compared with that of unrefined Al-Cu-Mn alloy, the tensile strength of refined alloy decreases more slowly as the temperature increases. Then the tensile strength of Al-Cu-Mn alloy with 0.3wt% Al-5Ti-0.8C master alloy increases by 7.1%, 8.7%, 13.5%, 14.6% and

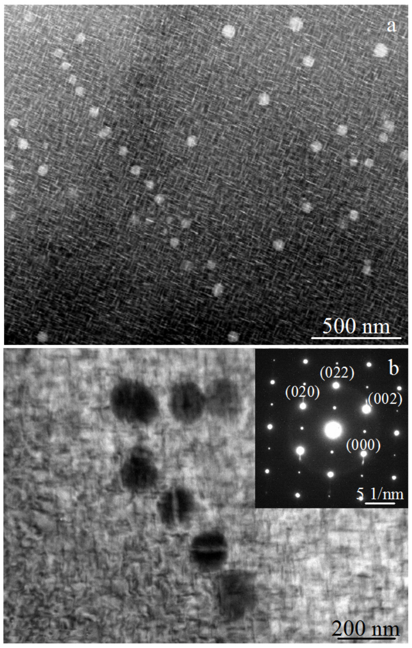

Fig.8 TEM images of Al_3(Zr, Ti) phases at low (a) and high (b) magnification

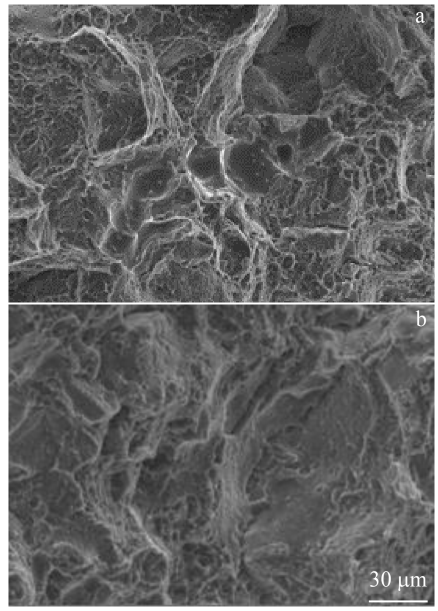

Fig.10 Tensile fractographies of Al-Cu-Mn alloy refined by different contents of Al-5Ti-0.8C master alloy: (a) 0wt% and (b) 0.3wt%

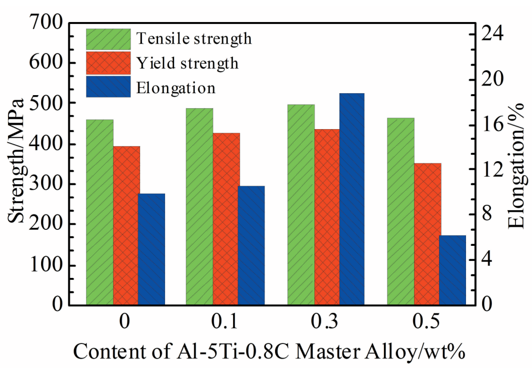

Fig.9 Tensile properties of Al-Cu-Mn alloys refined by different contents of Al-5Ti-0.8C master alloy after heat treatment

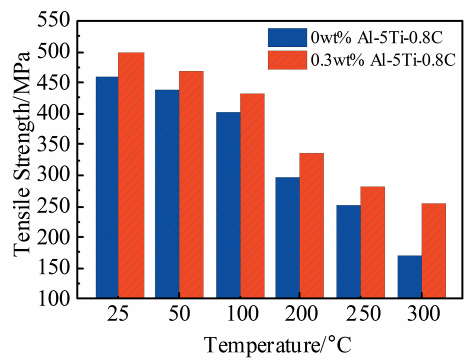

Fig.11 Tensile strength of Al-Cu-Mn alloy refined by 0wt% and 0.3wt% Al-5Ti-0.8C master alloy at elevated temperatures

50.3% at 50, 100, 200, 250 and 300 °C, respectively, indicating that the addition of Al-5Ti-0.8C master alloy improves the tensile strength of Al-Cu-Mn alloy at elevated temperatures.

According to the effect of Al-5Ti-0.8C master alloy and heat treatment on the grain size and microstructure, the better mechanical properties of Al-Cu-Mn alloy at elevated temperatures can be attributed to the finer grain size, higher fraction of θ'(Al_2Cu) precipitates, and the Al_3(Zr, Ti) precipitates.

Based on the Hall-Petch relationship, the improved strength $\Delta\sigma_{\text{Hall-Petch}}$ by the fine grain size can be expressed as Eq.(1)[30] as follows:

$$\Delta\sigma_{\text{Hall-Petch}}=k(d_{0.3\text{wt\% Al-Ti-C}}^{-1/2}-d_{\text{Al-Cu-Mn}}^{-1/2}) \quad (1)$$

where k is the Hall-Petch coefficient; $d_{0.3\text{wt\% Al-Ti-C}}$ and $d_{\text{Al-Cu-Mn}}$ are the average grain size of Al-Cu-Mn alloy with and without 0.3wt% Al-Ti-C master alloy addition, respectively. As shown in Fig. 3, the Al-5Ti-0.8C master alloy exhibits an excellent grain refining effect on the Al-Cu-Mn alloy, especially the alloy refined by 0.3wt% Al-5Ti-0.8C master alloy. Besides, the homogeneous distribution of the second phases of the refined Al-Cu-Mn alloy is also beneficial to the improvement of mechanical properties. Fig. 5 shows that the second phases are distributed uniformly in the matrix after refinement by Al-5Ti-0.8C master alloy, which is consistent with the conclusions of Stolyarova[31]. Thus, the refined Al-Cu-Mn alloy are strengthened considerably.

In addition, the new kind of Al-5Ti-0.8C master alloy promotes the formation of θ'(Al_2Cu) precipitates. Uniform and fine precipitates play an important role in hindering the movement of dislocations[32,33]. According to the Orowan mechanism of dislocation movement, the strengthening effect of precipitates can be expressed by Eq.(2~4)[34] as follows:

$$\Delta\sigma_{OR} = M\frac{0.4Gb}{\pi\lambda}\frac{\ln(\frac{2r}{b})}{\sqrt{1-v}} \quad (2)$$

$$\lambda = \left[(\frac{\pi}{f_p})^{\frac{1}{2}} - 2\right]r \quad (3)$$

$$r = \frac{d_p}{\sqrt{6}} \quad (4)$$

where $\Delta\sigma_{OR}$ is the increment in strength, and the meaning and values of other symbols are shown in Table 1.

Accordingly, the increments of Hall-Petch and Orowan strengthening due to the grain refinement and θ' (Al_2Cu) precipitates are calculated, as listed in Table 2. It can be seen that the improvement of mechanical properties for Al-Cu-Mn alloy refined by 0.3wt% Al-Ti-C master alloy is mainly attributed to the θ'(Al_2Cu) precipitates.

Furthermore, the Al-Cu-Mn alloy is refined by the spherical Al_3(Zr, Ti) precipitates after heat treatment, and some of precipitates present white lines in the middle (Fig.8), which is interpreted as anti-phase boundaries (APB)[40], indicating the phase transformation from metastable L12 to stable D023 structure. The generation of spherical Al_3(Zr, Ti) precipitates between the dendrites with low concentration and the nucleation and growth of precipitates are restricted by low driving force, which reduces the energy barrier of nucleation and inhibits the growth of dendrites. Therefore, the anti-aging ability and stability improve at high temperatures.

Table 1 Parameters in Eq.(1~4) for theoretical strength calculation of Al-Cu-Mn alloys

Symbol	Meaning	Value	Ref.
k/MPa·μm$^{1/2}$	Hall-Petch coefficient at 300 °C	22.4	[35]
$d_{Al\text{-}Cu\text{-}Mn}$/μm	Average grain size	100	-
$d_{0.3wt\%Al\text{-}Ti\text{-}C}$/μm	Average grain size	50	-
M	Orient factor of Al matrix	3.06	[36]
G/MPa	Shear modulus of Al matrix	2.54×10^4	[37]
b/μm	Burgers factor of Al matrix	2.86×10^{-4}	[38]
λ	Spacing of reinforcement	-	-
r/μm	Thickness of reinforcement	-	-
v	Poisson ratio	0.345	[39]
f_p/%	Volume fraction of reinforcement	1.09	-
d_p/μm	Average particle size of reinforcement	3×10^4	-

Table 2 Calculated strength increment for Al-Cu-Mn alloy with 0.3wt% Al-Ti-C master alloy

Temperature/°C	$\Delta\sigma_{HP}$/MPa	$\Delta\sigma_{OR}$/MPa
300	0.92	78.17

3 Conclusions

1) A new kind of Al-5Ti-0.8C master alloy has been fabricated using the self-propagating combustion reaction method and dilution treatment. The new master alloy contains α-Al, plate-like Al_3Ti, and fine granular TiC.

2) Al-5Ti-0.8C master alloy has an excellent grain refinement effect on Al-Cu-Mn alloys. With the increase of the master alloy content, the grain refinement effect firstly improves and then degenerates, and the morphology of θ' (Al_2Cu) phase changes from reticular to stripe shape. The Al-5Ti-0.8C master alloy facilitates the formation of θ'(Al_2Cu) precipitates during heat treatment.

3) Al-5Ti-0.8C master alloy improves the mechanical properties of Al-Cu-Mn alloys at room temperature and elevated temperatures, which is attributed to the finer equiaxed grain, the more dispersed distribution of second phases, and the larger amount of fine θ'(Al_2Cu) precipitates and Al_3(Zr, Ti) nanoparticles.

4) The microstructure and mechanical properties of Al-Cu-Mn alloys with 0.3wt% Al-5Ti-0.8C master alloy are optimal.

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新型Al-5Ti-0.8C中间合金对Al-Cu-Mn合金组织形貌和高温力学性能的影响

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摘　要：采用自蔓延燃烧反应法制备了一种新型Al-5Ti-0.8C中间合金，在此基础上采用不同含量（0%，0.1%，0.3%，0.5%，质量分数，下同）的中间合金对Al-Cu-Mn合金进行变质处理，研究该中间合金及其含量对Al-Cu-Mn合金组织形貌和高温力学性能的影响。结果表明：新型Al-5Ti-0.8C中间合金能够显著细化Al-Cu-Mn合金的晶粒尺寸，提高合金热处理过程中$\theta'(Al_2Cu)$相的析出密度，且细化析出相尺寸。其次，变质处理后合金的高温抗拉伸强度显著提高，且随着温度升高，抗拉伸强度的下降程度减小，主要原因在于变质处理后合金中析出均匀分布的细小$\theta'(Al_2Cu)$相以及热稳定性高的$Al_3(Ti, Zr)$纳米颗粒。此外，当Al-5Ti-0.8C中间合金含量为0.3%时，Al-Cu-Mn合金的组织形貌和高温力学性能最优。

关键词：Al-5Ti-0.8C中间合金；Al-Cu-Mn合金；晶粒细化；析出强化；高温力学性能

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