Effects of minor Y, Zr and Er addition on microstructure and properties of Al-5Cu-0.4Mn alloy

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Abstract: The effect of different contents of Y, Zr and Er on microstructure and properties of Al-5Cu-0.4Mn alloy was investigated. T6 heat treatment, OM, SEM and EDS methods were applied to the alloy. The results showed that fluidity and elongation of alloy adding Y, Zr and Er were improved, while with the increase of addition amounts, θ phase increased and grains were trended to grow up gradually. The Al-5Cu-0.4Mn alloy presented the maxed style of ductile and brittle fracture. After T6 heat treatment, the precipitation amounts of θ phase decreased dramatically and tensile strength and hardness significantly increased. Especially when addition contents were among 0.1–0.3wt.%, tensile strength and hardness of heat-treated alloy increased greatly, almost doubled as that of the as-cast state. The tensile strength reached its maximum of 378.43 MPa when the addition amount was 0.3wt.%. With the further increase of addition amounts, the elongation deteriorated and the proportion of ductile fracture reduced due to the limited dispersion strengthening effect of θ phase and $\text{Al}_6\text{Cu}_4\text{Er}$. It demonstrated that addition of 0.1–0.3wt.% Y, Zr and Er would generate positive effects and influences on Al-5Cu-0.4Mn alloy, which is significant for optimizing components and improving properties of Al-5Cu-0.4Mn alloy.

Key words: Al-Cu-Mn alloy; T6 heat treatment; microstructure; mechanical properties

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In recent years, with the continuous development of industrial technology, the demand of lightweight components is increasing, especially in the fields of aerospace, automobile, and boat moorings. Thus, the light weight, high strength Al-Cu-Mn alloys have considerable development prospects^[1-2]. However, the wide application of the alloy has been limited to a certain extent due to its casting defects, such as porosity, shrinkage and thermal cracking. In the process of improving the microstructure and properties of aluminum alloys, micro alloying has attracted many researchers' attention, and it is one of the most effective methods to control microstructure and obtain excellent comprehensive properties of metal alloys^[3-6].

Research shows that micro alloying elements not only has the purification function to aluminum alloy and reduces the content of high melting point phases, but also decreases the viscosity of aluminum alloy melt and increases fluidity, which has significant effects

on the mechanical properties of aluminum alloy^[7-8]. Among those alloying elements, adding element Y to aluminum alloy can prompt the spheroidization of the secondary phase of the as-cast alloy. At the same time, grain size is refined and the mechanical properties of the alloy are enhanced^[9-10]. Zr also has the positive effect on improving the properties of aluminum alloys. It can replace some lattice atoms of alloys, and trace Zr can significantly refine the alloy grain and inhibit recrystallization. For instance, with Zr addition, the microstructure of 2519 aluminum alloy can be refined and the comprehensive mechanical properties of Al-Mn-Si-Zn alloy can be improved^[11]. Al-based alloy matrix is strengthened as a result of Zr addition, which has a great effect on the improvement of microstructure and properties of the alloy^[12-17]. Second phase particles Al_3Er are formed by adding element Er in Al-Mg-Mn-Zr alloy, which can effectively pin dislocations. Meanwhile, grains of the alloy were refined and the thermal stability of the Al-5Mg alloy added Er was also improved^[18-19].

The effect of adding single or multiplex elements on microstructure and properties of aluminum alloy is different. Whereas, deposition strengthening and dispersion strengthening caused by multiplex elements are superior to that of a single one^[20]. Al-Cu-Mn alloy

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has a large crystal temperature range that causes defects such as segregation, shrinkage holes and hot cracking tendency during the casting process and leads to severe intergranular stress corrosion. Micro alloying elements play positive roles on improving the microstructure and properties of aluminum alloy, but research on properties of Al-5Cu-0.4Mn alloy with multiplex elements was barely reported. In this study, the change of microstructure and properties of Al-5Cu-0.4Mn alloy were investigated by adding Y, Zr and Er elements, which would provide a theoretical basis for the further improvement of the alloy performances.

1 Experimental procedure

Al-5Cu-0.4Mn alloy was used as raw material (chemical composition is shown in Table 1) and different amounts of Y, Zr and Er (0, 0.1, 0.3 and 0.5wt.%, respectively) were added to the alloy, as shown in Table 2. The raw alloy was added when the furnace temperature was 400–450 °C, and 0.5% C₂Cl₆ was

added with a bell jar to refine the base alloy for 10 min at 730±5 °C. The Al-10.70wt.%Y, Al-10.60wt.%Zr and Al-12.61wt.%Er intermediate alloys were added into the crucible furnace at 750±5 °C. The cylindrical samples for fluidity and mechanical properties testing (length: 130 mm, diameter: 13 mm) were cast in metal molds when the temperature of the liquid metal was 730±5 °C. Some of the samples were treated with T6 technology (540 °C×10 h for solid solution and 150 °C×8 h for artificial aging treatment). The samples for microstructure observations were polished and corroded by HF liquid (99.5%H₂O + 0.5%HF). The microstructure morphologies were observed by GSM-6700F scanning electron microscopy (SEM), and the phase composition was confirmed by Energy dispersive X-ray spectroscopy (EDS). The tensile test (sample is shown in Fig. 1) was conducted on an AG-10TA universal electronic tensile testing machine, and Vickers hardness was also measured. Finally, comprehensive theoretical analysis on microstructure and properties of the alloy after adding alloying elements was conducted.

Table 1: Chemical composition of Al-5Cu-0.4Mn alloy (wt.%)

Cu	Mn	Ti	Cd	V	Zr	B	Al
5	0.4	0.2	0.2	0.25	0.2	0.05	Bal.

Table 2: Addition amount of intermediate alloys

Furnace No.	1	2	3	4
Y+Er+Zr (wt.%)	0	0.1	0.3	0.5
Al-10.70wt.%Y (g)	0	8.4	28.1	42.1
Al-10.60wt.%Zr (g)	0	8.5	28.3	42.5
Al-12.61wt.%Er (g)	0	9.5	23.8	47.6
Al-5Cu-0.4Mn (g)	3000	2973.6	2919.8	2867.8

Fig. 1: Size of tensile sample

2 Results and discussion

2.1 Effect of alloying elements on microstructure

The microstructures of the as-cast alloy with 0, 0.1, 0.3 and 0.5wt.% alloying elements are shown in Fig. 2. As can be seen from Fig. 2, with the increase of the amount of alloying elements, a large amount of precipitation θ phase emerged.

Figure 2(b) shows that the amount of the θ phase in the matrix (as indicated by red arrow) is more than that of the alloy without alloying element and shows strip shape, while small parts distribute as mesh structure. When the alloying element content arrives at 0.3%, as shown in Fig. 2(c), the number and size of θ phase further increase, but the uniformity of the distribution is reduced, and the grain size is larger than the one shown in Fig. 2(b). When the addition content further increases to 0.5wt.%, the number of θ phase is decreased, while the shape and size of the particles changed little.

Figure 3(a)–(d) shows the microstructure morphologies of T6 treated alloy with different amounts of alloying elements. It can be seen that the number of the θ phase is decreased and the shape changes to spotted-like compared with the as-cast ones. As shown in Fig. 3(b)–(d), with the increase of addition contents of alloying elements, the θ phase precipitated from the matrix increases gradually in quantity and distributes more dispersedly, but the grain shape is not significantly changed. When the addition amount reaches 0.5wt.%, the amount of precipitated θ phase further increases, and reticular structures appear [as indicated by the red arrow in Fig. 3(d)], which are unfavorable to uniformity of the alloy microstructure.

EDS results of the as-cast and heat treated alloys containing 0.3wt.% Y, Zr and Er elements are shown in Fig. 4 and Table 3. It can be found that the matrix microstructure of the as-cast alloy mainly consists of Al and Cu, and the contents of Y and Er elements on the grain boundaries (GBs) are more than those in the matrix. It indicated that most of the alloying elements remain at GBs. In addition to θ phase, other phases related to Y, Er and Zr are formed. While after heat treatment, the content of

Fig. 2: Microstructures of as-cast alloy with different addition contents of Y, Zr and Er: (a) 0, (b) 0.1wt.%, (c) 0.3wt.% and (d) 0.5wt.%

Fig. 3: Microstructures of T6 heat treated alloy with different addition contents of Y, Zr and Er: (a) 0, (b) 0.1wt.%, (c) 0.3wt.% and (d) 0.5wt.%

Y is slightly increased, Er and Zr amounts are decreased in the matrix of the alloy, and the content of Al, Cu, Y, Er and Zr at GBs is decreased.

It was considered through analysis that the solid solubility of added elements Y, Er and Zr in matrix α -Al is poor. During the cooling and solidification process of the alloy, substitutional solid solution formed due to the diameter difference between the matrix atoms and added Y, Er and Zr (Al: 0.143 nm, Y:

0.18 nm, Er: 0.245 nm, Zr: 0.216 nm). As a result, dislocation density and migration resistance of GBs increase. Meanwhile, θ phase, Al_3Zr , Al_3Y , etc. and part of the undissolved alloying elements enriched on the solid-liquid interface increased the undercooling degree and therefore increased the particles' heterogeneous nucleation, which is beneficial to the grain refinement^[21-23]. However, when the addition amount reached 0.3%, too many solute atoms enriched on GBs and vicinity

Fig. 4: SEM and local EDS images of alloy with 0.1wt.% Y, Zr and Er: (a)–(c) as-cast, (d)–(f) T6 heat treated

Table 3: Elements distribution of as-cast and T6 heat treated alloy with 0.1wt.% Y, Zr and Er

Conditions	Element content (wt.%)	Al	Cu	Y	Zr	Er
As-cast	Position A (matrix)	88.75	4.75	0.4	1.02	1.29
	Position B (grain boundary)	57.70	10.55	1.75	0.74	9.00
T6 heat treatment	Position C (matrix)	84.55	1.36	0.46	0.37	0.66
	Position D (grain boundary)	57.67	28.19	0.02	0.05	0.51

areas, so the heterogeneous nucleation process was weakened and the grain size increased slowly, θ phase and alloying elements were blocked on GBs. With the increase of Y, Er and Zr contents, Al_3Zr and other particles were coarsening, which resulted in an adverse effect on the uniformity of the microstructure^[24-27].

After T6 treatment, the number of θ phase and Y, Er and Zr elements distributed in the matrix and GBs reduced, which was associated with the solid solution diffusion and aging degree of the alloy during heat treatment. With the solution treatment carrying on, θ phase continuously dissolved into the matrix $\alpha\text{-Al}$. Al_3Er , Al_3Zr , Al_3Y , etc., and undissolved alloy elements gathered on GBs and its vicinity began to move and fully diffuse, and local segregation was eliminated. However, due to the formation of particles such as $\text{Al}_8\text{Cu}_4\text{Er}$ and AlCuY , some copper atoms were consumed that made the number of θ phase continuously reduced and supersaturated solid solution did not occur in this process. In the subsequent aging process, the amount of the precipitation of θ phase reduced. Therefore, the number of θ phase in T6 treated alloy was greatly reduced and tended to be in dispersive distribution, which would play an important role in improving the microstructure and properties of the studied alloy.

2.2 Effects of alloying elements on properties of alloy

2.2.1 Effect on fluidity

The change of alloy fluidity is shown in Fig. 5. It can be seen that the fluidity improves greatly after Y, Zr and Er were added. When the addition amount is 0.1wt.%, the fluidity increases from 116.67 mm to its maximum value of 272.67 mm. After that moment, with the increase of the addition amounts, the fluidity value tends to come down and basically maintains at about 230 mm. After adding 0.5wt.% of the alloying elements, the fluidity began to slowly increase to 251 mm. This demonstrates that adding alloying elements can improve the fluidity of Al-Cu-Mn alloy. The chemical properties of Y and Er elements are more active and have strong affinity with elements in the alloy, adsorbing impurity atoms and oxide inclusions Al_2O_3 easily. The reaction process is as Eq. (1) and (2):

The oxidation inclusion Al_2O_3 was reduced to Al, shrinkage cavity and slag inclusions were decreased to a certain extent due to the high activity of Y and Er elements. Therefore, addition of Y and Er has the purification function to degassing and slag removal. Surface tension and viscosity of alloy melt were also

Fig. 5: Fluidity curve of Al-Cu-Mn alloy with different Y, Zr and Er contents

reduced. At the same time, the temperature interval of liquid to solid transition was reduced. The layer by layer solidification style was enhanced and the feeding capacity was improved in the filling process. Therefore, after adding Y, Er and Zr, the fluidity of the alloy was greatly improved.

2.2.2 Effect on mechanical properties and hardness

The mechanical properties and hardness curves of the alloy

containing Y, Zr and Er at as-cast and T6 treated status are shown in Fig. 6. As can be seen from Fig. 6, with the increase of the added elements, the elongation of the as-cast alloy is maintained at a rising state, increasing from the original value of 9.07 mm to 15.13 mm. While the tensile strength of as-cast alloy falls to 197.34 MPa when the addition content is 0.1wt.%, and slowly increases to 204.84 MPa when the adding amount is 0.3wt.%. There is no obvious change in tensile strength when the amount of alloying elements is further increased. The hardness decreases slightly when the addition content is 0.1wt.%, and is basically not changed and maintains at about 56 HV when alloying elements are further added to 0.5wt.%. After heat treatment, the tensile strength and hardness of the alloy are greatly improved, almost doubled that of the as-cast state, while the elongation decreased. With the increase of the addition amounts, the tensile strength and elongation shows a trend of first increase and then decrease. When the adding amount is 0.1wt.%, the elongation decreases to 2.16%. The maximum values of tensile strength and elongation are obtained at 0.3wt.% addition, which are 378.43 MPa and 6.9%, respectively. After that point, the hardness value reduces from its original value of 122 HV to 87.7 HV.

Fig. 6: Properties curves of Al-Cu-Mn alloy containing different Y, Zr and Er contents: (a) tensile strength, (b) elongation and (c) hardness

Figure 7 (a)-(c) shows that the eutectic reaction temperature of Al-Y, Al-Er and Al-Zr is: 645 °C, 655 °C and 660 °C, respectively. It also can be seen from the Al-Cu-Mn ternary alloy phase diagram shown in Fig. 7(d) that T_1 ($\text{Al}_{12}\text{Mn}_2\text{Cu}$) was transformed to (Al)- θ phase in solidification process. In the cooling and solidification process, the added Zr element firstly reacted with α -Al, and the formed Al_3Zr particles can be used as the nucleus, and the nucleation rate of the alloy liquid is increased. But with the decrease of temperature, eutectic reaction of Al-Y and Al-Er could occur one after another. Among them, the interactions between Er, Al and Cu occurred and generated the low melting point eutectic compounds $\text{Al}_8\text{Cu}_4\text{Er}$ and AlCuY particles near the GBs that lower grain continuity and deformation coordination ability of grains were reduced. As result, the strength near GBs was limited and caused the strength of the alloy reduced. Meanwhile, as the nucleus, these particles and dissolved Y, Er and Zr elements hindered the diffusion and movement of Cu atoms and θ phase. With increasing the amount of the alloying elements, due to the limited solubility and most of the added elements segregated

on the GBs, the result is that the GB segregation increased and the diffusion channels of the dendrites were blocked, which caused most of the Cu atoms and θ phase stopped on the GBs. Therefore, lots of θ phase precipitated out in the matrix, which deteriorated the uniformity of the microstructure and decreased the tensile strength and hardness values.

However, θ phase, Al_3Y , Al_3Er and Al_3Zr generated at the same time due to the short temperature interval of the three eutectic reactions and closed temperature between Al-Cu binary alloy reaction and those eutectic reactions, the small radius of $\text{Al}_3(\text{Y, Er, Zr})$ particles also formed, which played an important role in improving the properties of the Al-5Cu-0.4Mn alloy. In addition, mutual competition between added alloying elements and matrix atoms took place because of their similar reaction temperatures, which could reduce the segregation behavior caused by undissolved alloy elements. The generated Al_3Y , Al_3Er and Al_3Zr had a dispersion strengthening effect on the studied alloy. The amount of θ phase was little changed, thus, the tensile strength and hardness of the as-cast alloys were not changed obviously, while the elongation

Fig. 7: Alloy phase diagrams: (a) Al-Y, (b) Al-Er, (c) Al-Zr and (d) Al-Cu-Mn alloy

kept a tendency to keep rising slowly when the addition amount was more than 0.3wt.%.

The increase of tensile strength and hardness of the alloy after heat treatment was caused by the elimination of the micro-segregation and the microstructure trend to distribute uniformly. What's more, with the increase of addition amount of the alloying elements, the number of Al_3Y , Al_3Zr , $\text{Al}_8\text{Cu}_4\text{Er}$, AlCuY and other intermetallic compound particles increased and inevitably tended to segregated on GBs and the nearby areas. The activation energy of the alloy under T6 treatment (540 °C) was not enough for a large number of Y, Er and Zr elements to diffuse completely, so that the elements could not come into the center parts of the alloy at the end of the solid solution treatment. The concentration difference between the matrix and its edge locations was not completely eliminated. Last but not least, some θ phases were blocked at GBs during the solidification process. As a result, the number of θ phase dissolving into the matrix was not enough for the alloy to develop a supersaturated solid solution. So the precipitation number of the θ'' and θ in the aging process was reduced and the strengthening effect on alloy matrix was weakened. At the same time, the dispersion of Al_3Zr particles increased, and as a result, the resistance of dislocation movement increased. Therefore, after adding more than 0.3wt.% alloying elements, the tensile strength and hardness of the alloy appeared to decline and the elongation rate increased slowly under the influence of the above three factors when the addition amount was more than 0.3wt.%.

2.2.3 Effect on fracture behavior

The fracture properties of alloys with Y, Er and Zr addition (0.1 and 0.3wt.%) before and after heat treatment were analyzed. Fig. 8 (a)–(b) shows that the fracture morphologies of the alloy present a large number of different shapes and sizes of the equiaxed dimple structure, and river appearance structure and tear ridges also can be seen on fracture surfaces when the addition amount is 0.1wt.%, indicating that the alloy has mixed failure modes of ductile and brittle fracture. When the adding content is 0.3wt.%, the size and number of dimples are reduced, cleavage steps and a small amount of micro cavities appear, which demonstrate that the ratio of ductile fracture of the alloy is decreased. After heat treatment, as shown in Figs. 8 (c) and (d) for added 0.1wt.% alloying elements, in addition to the small size of the dimple structure, the fracture morphologies of the alloy take on many irregular cleavage steps. When contained content is 0.3wt.%, the structure of gully appearance is found, the numbers of shear lips and dimples are further reduced, and there are a certain number of holes and cracks. What can be learned is that ductile fracture is the main destruction mode of as-cast alloy. With the increase of element adding contents, the ratio of ductile fracture decreases, while the alloy presents fracture failure in the style of a mixed mode (brittle fracture and cleavage fracture) after heat treatment.

Analysis suggests that the stomata and impurity elements are adsorbed to matrix and near the GBs due to the addition of alloying elements in the solidification process, so that the

Fig. 8: SEM fracture morphologies of alloy with different Y, Zr and Er contents: (a) and (b) are fracture morphologies of as-cast alloy with 0.1 and 0.3wt.% Y, Zr and Er, (c) and (d) are fracture morphologies of T6 treated alloy with 0.1 and 0.3wt.% Y, Zr and Er

micro crack source can be eliminated to a certain extent. At the same time, the new generated intermetallic compounds and undissolved alloying elements block the dislocations motion, resulting in suppression of the cracks propagation, therefore, the alloy mainly showed the failure mode of ductile fracture. However, due to the influence of atomic size, lattice distortion emerged inevitably in the process of alloying elements dissolving into the α -Al matrix, after solidification, which will lead to the existence and accumulation of a low stress state. Under external stress, initiation of micro cracks first took place along the tip parts of θ phase particles and intersections of θ phase and slip belts. And cracks propagated and expanded along GBs with segregations and the parts where the brittle $\text{Al}_8\text{Cu}_4\text{Er}$, AlCuY and coarsened Al_3Zr particle precipitated. The movement of cracks propagating to GBs was hindered and new micro cracks would form in cleavage surfaces, the time of cracks mutually connected with each other and merged into a whole was delayed to a certain extent. So finally, the alloy presented the major failure mode of ductile fracture.

After T6 treatment, the precipitation number of θ phase was decreased and precipitate-free zones (PFZ) around GBs were broadened. As a result, GBs strength was reduced and dislocation pile-up and stress concentration exacerbated. The probability of cracks formed in large second phases was greatly increased. Especially when the adding amount was more than 0.3wt.%, low melting point AlCuY particles and eutectic compound $\text{Al}_8\text{Cu}_4\text{Er}$ on GBs further increased and plasticity of the alloy reduced^[28]. The deformation coordination ability between grains was abated and the ratio of brittle fracture of the alloy further increased.

It indicates that Y, Er and Zr have important effects on the improvement of the properties of Al-Cu-Mn alloy. Combined with the experimental phenomenon and results above, the adding amount should be adjusted between 0.1–0.3wt.%. The more rational content of each element still needs more studies and experiments.

3 Conclusions

(1) After adding elements Y, Zr and Er, the as-cast alloy microstructure was refined. Especially when 0.1wt.% alloying elements was added, the effect of grain refinement was the best and θ phase distributed in strip or puncta form. When adding amounts were more than 0.3wt.%, the number of precipitates increased and changed to grid structure. After T6 treatment, the amount of precipitates was greatly reduced and distributed dispersedly, while the grain size was little changed.

(2) The fluidity was greatly improved and reached the highest value of 272.67 mm when adding amount of the alloying elements was 0.1wt.%.

(3) The tensile strength and hardness of the as-casting alloy were slightly decreased, while the elongation was increased with addition of alloying elements. After T6 heat treatment, the tensile strength and hardness were greatly improved, and the elongation was decreased. Among them, the tensile strength showed the most significant increasing tendency and obtained a maximum value of 378.43 MPa when adding amounts were 0.1–0.3wt.%. Elongation also presented the trend of recovery.

(4) Due to the generation of $\text{Al}_8\text{Cu}_4\text{Er}$, AlCuY and other metal compounds, the number of the basic strengthening phase θ was

reduced to a certain extent. The alloy showed a mixture failure mode of ductile and brittle fracture, and ductile fracture was in dominant position when the adding amount was 0.1wt.%. The ratio of the brittle fracture of the alloy was increased after heat treatment.

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