Effects of Cu content on microstructure and mechanical properties of rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys

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Abstract: A systematic study on how Cu content affects the microstructure and mechanical properties of rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys during solution treatment and ageing heat treatment was conducted. The swirled enthalpy equilibrium device (SEED) was adopted to prepare the semi-solid slurry of Al-6Zn-2Mg-*x*Cu alloys. The microstructure development and mechanical properties were studied using optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC), as well as hardness and tensile testing. The grain boundary and shape factor were calculated using image processing software (Image-Pro Plus 6.0). Results show that the alloys are composed of typical globular primary α-Al grains, eutectic phases, and smaller secondary α-Al grains. After solution and ageing heat treatment, the eutectic phases are dissolved into Al matrix when the Cu content is lower than 1.5wt.%, while some eutectic phases transform into Al_2CuMg (S) phases and remain at grain boundaries when Cu content reaches 2wt.%. T6 heat treatment significantly enhances the mechanical properties of rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys. When Cu concentration is 0.5wt.%–1.5wt.%, the ultimate tensile strength, yield strength and elongation of T6 treated alloys rise to around 500 MPa, 420 MPa, and 18%, respectively.

Keywords: Al-Zn-Mg-Cu alloy; rheo-diecasting; Cu content; mechanical properties; heat treatment

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1 Introduction

Despite the high cost and difficult production process, the 7xxx (Al-Zn-Mg-Cu) wrought aluminum alloys are still widely used in aerospace industry due to its well-performing mechanical qualities and superior fatigue resistance [1-3]. Recently, there has been a booming interest in producing cast Al-Zn-Mg-Cu alloys with the goal of lowering production costs and developing shaped products while maintaining outstanding mechanical qualities [4].

Several processes, such as spray deposition, electromagnetic casting, powder metallurgy, semi-solid casting and quick solidification, have been employed to solve the difficulties of producing near-net-shape castings [4]. Semi-solid die casting has been shown a reliable and efficient method for producing high-quality products in the automobile industry [5, 6]. The round and tiny microstructures generated during semi-solid slurring have been reported to improve the castability of the Al alloys in semi-solid die casting [7]. For example, using the high solid fraction semi-solid die casting method, 7xxx series alloys castings were effectively produced [8].

In addition to the casting properties of the alloy, the mechanical properties of the cast Al-Zn-Mg-Cu alloys are more important. The research works show that the existence of Cu element can improve the dispersion degree of precipitation phase, and further improve the strength and plasticity of Al-Zn-Mg-Cu alloys [9-11]. The effect of Cu content on the microstructure and characteristics of wrought alloys has been well studied. However, it is still unclear how the Cu influences the mechanical characteristics of semi-solid die casting Al-Zn-Mg-Cu alloys. Therefore, it is necessary to systematically study the effect of different contents of

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Cu on the microstructure and properties of semi-solid casting Al-Zn-Mg-Cu aluminum alloys, so as to obtain alloys with higher mechanical properties. In this study, Al-6Zn-2Mg-*x*Cu alloys were selected as the research object and the evolution law of the alloy microstructure and properties was obtained by changing the content of Cu and heat treatment process.

2 Experimental procedure

Table 1 lists the chemical composition of the Al-Zn-Mg-*x*Cu alloys, which were prepared by a combination of pure Al, Zn, Mg, and master alloys Al-50Cu (wt.%), Al-5Ti-B (wt.%) (Note: The actual content of Cu in the Al-6Zn-2Mg-2Cu alloy differs from the nominal composition due to the composition deviation of the Al-50Cu master alloy, however this has no effect on the Cu change trend in the study). Firstly, pure Al and Al-50Cu alloys were melted at 720 °C in an electrical resistance furnace. Then, pure Mg and Zn were added at 680 °C. The melt was then heated to 750 °C, and Al-5Ti-1B rods were added. With the argon protection, a rotating impeller was used for degassing. Secondly, the semi-solid slurry was produced by using the swirled enthalpy equilibrium device (SEED): the liquid melt was poured into a steel crucible at 650 °C. The melt was cooled to about 617 °C in a concentric cylinder rotated at a speed of 180 rpm. Thirdly, the samples were made using a Buhler 340 t die casting machine. With an casting pressure of 950 bar and an filling speed of 2 $m \cdot s^{-1}$, the semi-solid slurry was transferred into the horizontal shot sleeve and injected into the die. Finally, the samples were heat treated for 8 h at 470 °C before being quenched with water to room temperature. The samples were then artificially aged at 120 °C for different times of 0.5 h to 192 h.

The microstructure of the alloy samples was studied using optical microscopy (OM) and scanning electron microscopy (SEM) with EDS. The shape factor and average diameter of the primary α-Al grains were calculated using image processing software (Image-Pro Plus 6.0). Using differential scanning calorimetry, a thermal study was carried out at a rate of 10 $°C \cdot min^{-1}$ (DSC). With an $8° \cdot min^{-1}$ scanning speed and a 2θ between 10° and 90°, XRD testing was performed to reveal the phases evolution of the alloys. The alloys' hardness was determined using Vickers hardness equipment with a load of 5 kg and a duration of 15 s. The universal tensile tester was used to test the tensile properties of the samples with 5 mm gauge in diameter and 25 mm gauge length at an initial strain rate of 1.6×10^{-4} s^{-1}.

Table 1: Chemical compositions of Al-6Zn-2Mg-*x*Cu alloys (wt.%)

Alloy	Zn	Mg	Cu	Fe	Si	Al
0Cu	6.17	2.10	<0.0002	<0.0001	0.0194	Bal.
0.5Cu	5.98	2.06	0.55	<0.0001	0.0066	Bal.
1Cu	6.26	1.98	1.16	<0.0001	<0.0003	Bal.
1.5Cu	5.82	1.83	1.53	<0.0001	<0.0003	Bal.
2Cu	6.10	1.99	2.46	<0.0001	<0.0003	Bal.

3 Results and discussion

The typical OM images of the as-cast Al-6Zn-2Mg-*x*Cu alloys are shown in Fig. 1. The OM figures show that all the experimental alloys contain uniform globular primary α-Al grains, smaller secondary α-Al grains and eutectic phases (grain boundary structure), which are identified by the arrows in the figures. The primary α-Al grains are formed during the slurry preparation process, while the secondary α-Al grains and eutectic phases are formed during the solidification process of

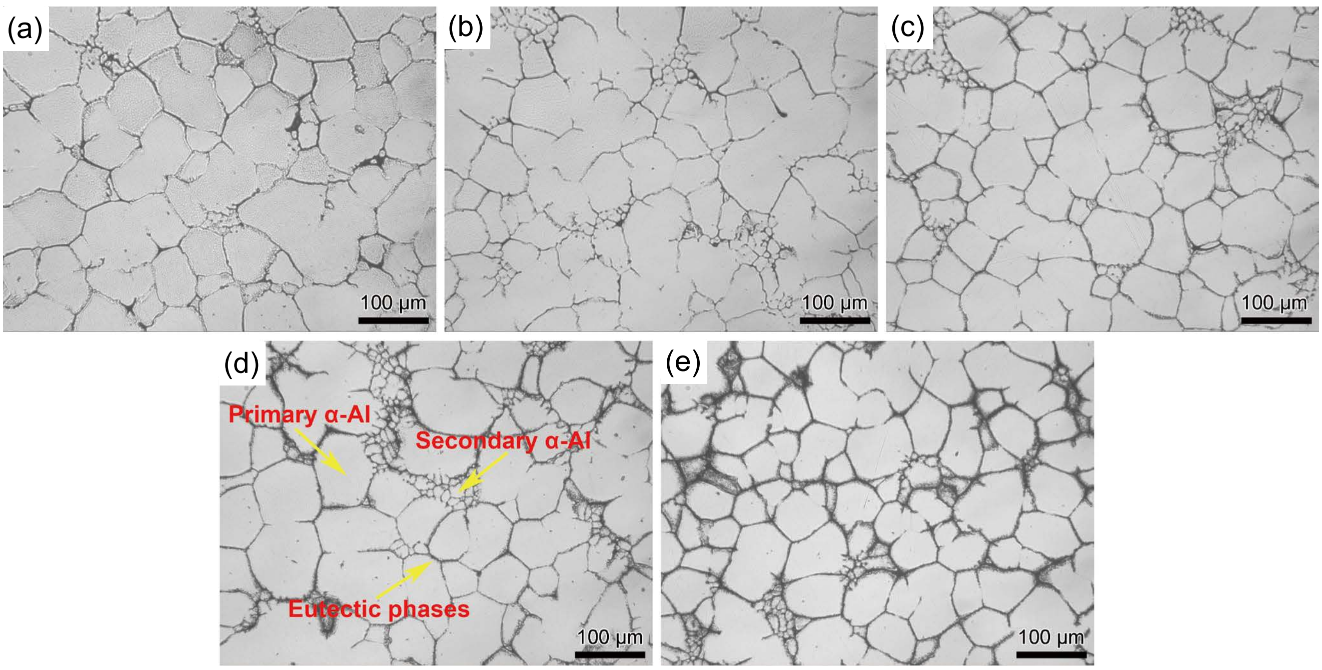


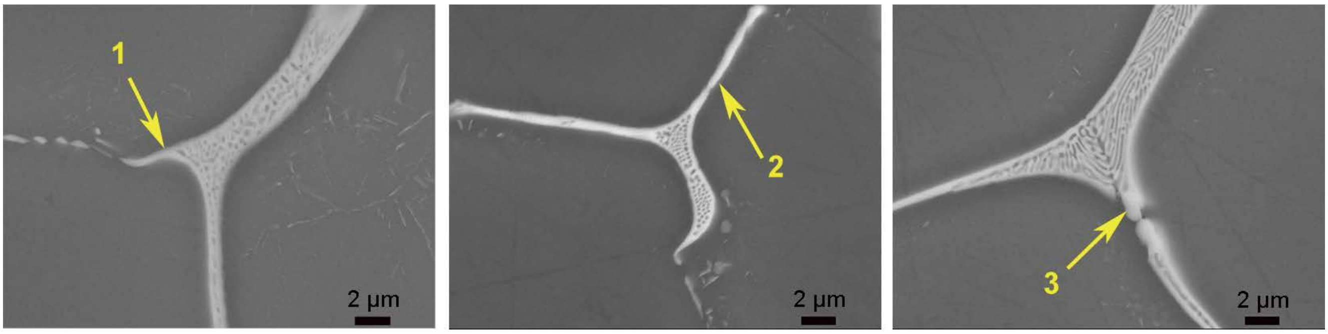
Fig. 1: OM images of as-cast Al-6Zn-2Mg-*x*Cu alloys: (a) 0Cu; (b) 0.5Cu; (c) 1Cu; (d) 1.5Cu; and (e) 2Cu

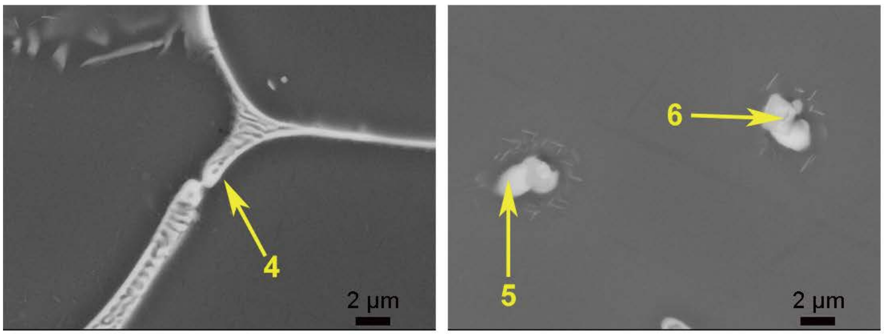
the die casting [12]. Also, Fig. 1 indicates that Oswald ripening or coalescence occurs during the grain growth.

Figure 2 shows the SEM images of intermetallic compounds distributed in the grain boundaries and grain phases, as well as the element analysis results. It shows that when the Cu content less than 1wt.%, Cu does not exist in the intermetallic particles. In addition, the different compositions of Points 5 and 6 indicate that the intermetallics are different in as-cast Al-6Zn-2Mg-2Cu alloy.

Figure 3 shows the XRD analysis of the Al-6Zn-2Mg-*x*Cu alloys. As the Cu content increases, the intensity of the diffraction peaks of the eutectic phase becomes stronger, which indicates that the content of eutectic phase in the as-cast microstructure increases with the increase of Cu content. The result also shows that the main second phase in the as-cast ingot is Mg(Al, Zn, Cu)$_2$. Some Zn atoms in $MgZn_2$ were replaced by Al and Cu atoms to form Mg(Al, Zn, Cu)$_2$. Besides, there is a small amount of AlZnMgCu phase in the as-cast samples when the alloys contain 0 and 0.5wt.% Cu. When Cu is less than 2wt.%, the intermetallic compounds in all as-solution samples are Mg(Al, Cu, Zn)$_2$. However, a new phase Al_2CuMg appears in the as-solution Al-6Zn-2Mg-2Cu alloy sample.

Figure 4 illustrates the size and shape factor of the grains in the as-cast Al-6Zn-2Mg-*x*Cu alloys, as well as the area fraction of eutectic phase. The change trend of average grain size and shape factor of the as-cast Al-6Zn-2Mg-*x*Cu alloys is not obvious





Point	Al	Zn	Mg	Cu
1	70.78	12.88	16.34	-
2	75.94	9.83	14.23	-
3	70.22	12.21	13.56	4.01
4	71.01	9.58	14.10	5.31
5	71.67	9.78	12.54	6.01
6	69.76	5.28	6.83	18.13

(f)

Fig. 2: SEM images of the second phases (intermetallic compounds) in as-cast Al-6Zn-2Mg-*x*Cu alloys and their composition analysis: (a) 0Cu; (b) 0.5Cu; (c) 1Cu; (d) 1.5Cu; (e) 2Cu; and (f) composition of Points 1 to 6 (at.%)

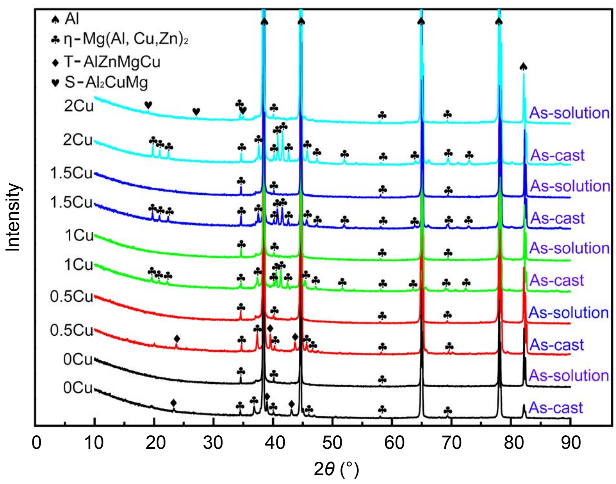


Fig. 3: XRD patterns of as-cast and solution treated Al-6Zn-2Mg-*x*Cu alloys

with the Cu content, and the average values of which are about 80 μm and 0.45, respectively, indicating that the content of Cu has little effect on grain refinement of the alloy. However, the area fraction of eutectic phases is increased from 2.8% to 6.4% when the Cu content is increased from 0 to 2wt.%.

The DSC tests in Fig. 5 show that an endothermic peak appears at about 478 °C for all the as-cast Al-6Zn-2Mg-*x*Cu alloys, which is attributed to the melting of the (Al+η) eutectic phases. Besides, another endothermic peak at about 486 °C is found for the as-cast Al-6Zn-2Mg-2Cu alloy. As mentioned above, Al_2CuMg phase will be precipitated in the as-solution sample when the Cu content increases to 2wt.%, which is also the main difference from the other alloys. Therefore, it is obvious that the second endothermic peak on the DSC curve of Al-6Zn-2Mg-2Cu alloy corresponds to the remelting temperature of Al_2CuMg.

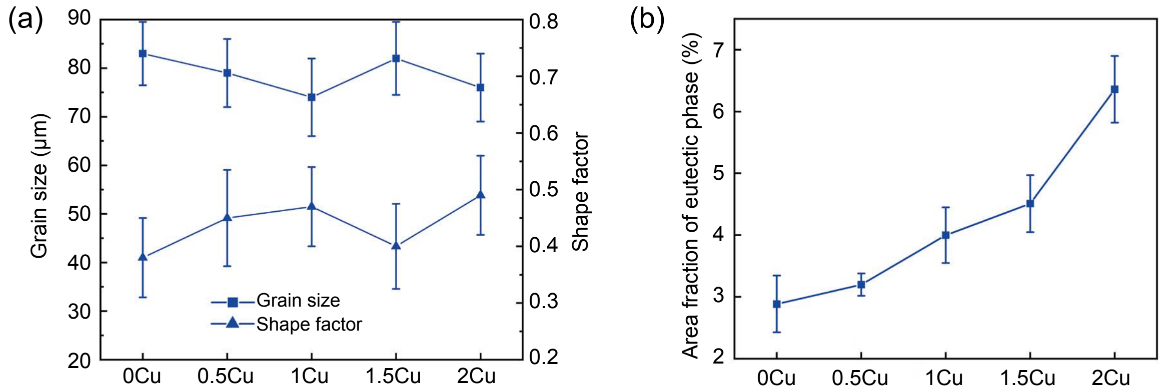


Fig. 4: Grain size, shape factor (a) and eutectic area fraction (b) of as-cast Al-6Zn-2Mg-*x*Cu alloys

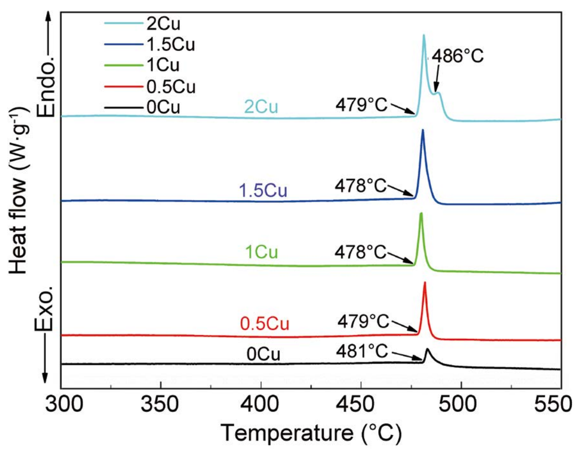


Fig. 5: DSC analysis of as-cast Al-6Zn-2Mg-*x*Cu alloys

The typical SEM images of solution-treated Al-6Zn-2Mg-*x*Cu alloys are shown in Fig. 6. For alloys with Cu content less than 1.5wt.%, most of the eutectic phases are dissolved into the Al matrix after solution treatment. However, some undissolved phases remain at grain boundaries when the Cu content reaches 2wt.%.

According to the solid solubility line of Al_2CuMg phase in Al-6Zn-*y*Mg-*x*Cu alloy that shown in Fig. 7 [13], the alloy with the Mg content of 2wt.% will be transformed into the single α-Al phase at about 470 °C when the Cu content is less than or equal to 1.5wt.%, while it consists of α-Al and Al_2CuMg phases when the Cu is 2wt.%. Therefore, it can be inferred that the remained phase in Fig. 6(e) is the Al_2CuMg, which is also confirmed by the results of SEM and EDS, as shown in Fig. 8. The XRD patterns of the solution-treated Al-6Zn-2Mg-*x*Cu alloys (Fig. 3) also prove the existence of Al_2CuMg phase (S) in solution-treated Al-6Zn-2Mg-2Cu alloy.

Previous studies have shown that most of T and η phases are dissolved into the matrix during solution treatment [14]. The S-Al_2CuMg phase adheres to the η-$MgZn_2$ phase to form nucleation, and then solid-state phase transition occurs. The period for S phase transformation is within 0–12 h after the start of solid solution, and its content reaches the peak value

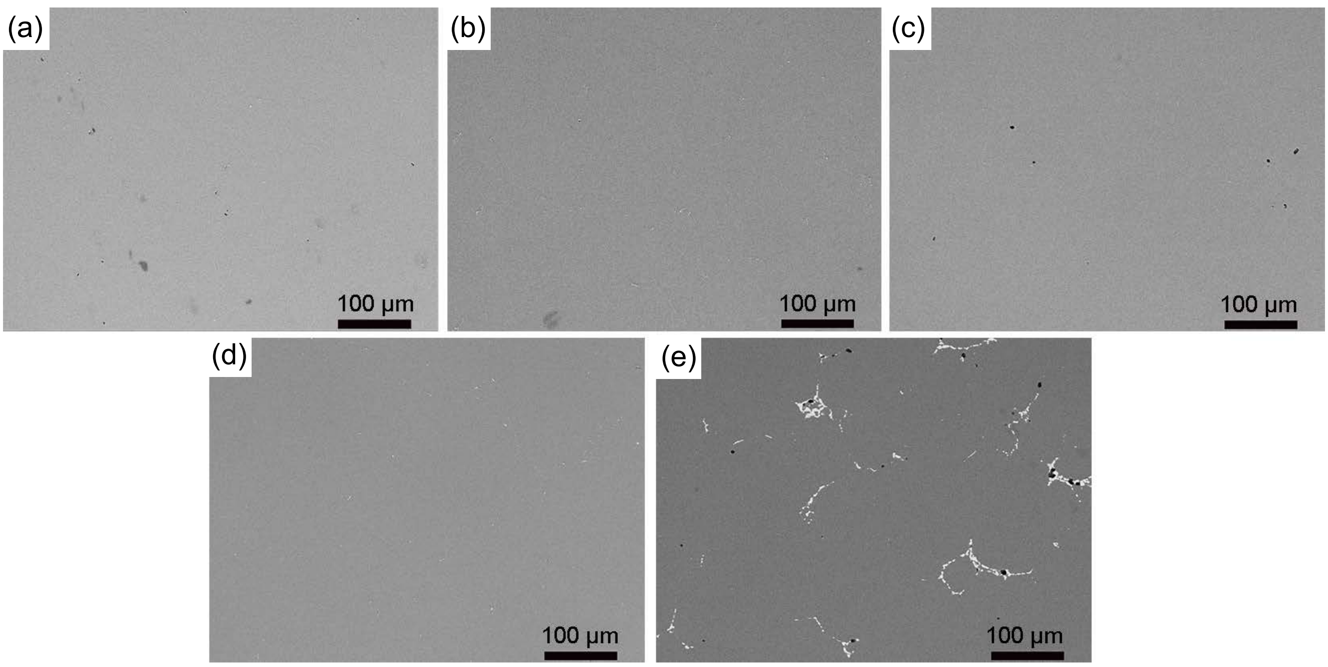


Fig. 6: SEM images of solution-treated Al-6Zn-2Mg-*x*Cu alloys: (a) 0Cu; (b) 0.5Cu; (c) 1Cu; (d) 1.5Cu; and (e) 2Cu

within 6 h [14]. Therefore, it is reasonable to believe that the alloy with 2wt.% Cu content can undergo phase transformation and form Al_2CuMg phase during solid solution process. It corroborates well with the SEM and DSC results that the undissolved phases in Fig. 6(e) and the endothermic peak around 486 °C in Fig. 5 refer to the Al_2CuMg phase.

The hardness evolution of solution-treated Al-6Zn-2Mg-*x*Cu alloys followed by isothermal ageing at 120 °C for different times is shown in Fig. 9. Note that all alloys exhibit the same hardening trend. It can be seen that the hardness rapidly increases within the initial 24 h, then slowly increases to the peak hardness at 128 h and remains stable even up to 192 h. The final hardness of all the alloys increases obviously, indicating that the hardness of the Al-6Zn-2Mg-*x*Cu alloy is sensitive to isothermal ageing. Furthermore, the hardness curves show that the hardness of the alloys increases with increasing the Cu content. The hardness of Al-6Zn-2Mg-2Cu alloy is obviously higher than the other Al-6Zn-2Mg-*x*Cu alloys, and the difference of hardness between the other Al-6Zn-2Mg-*x*Cu alloys is relatively small.

The mechanical properties of as-cast, solution-treated and solution-treated combined with aged (T6 for 24 h) Al-6Zn-2Mg-*x*Cu alloys are shown in Fig. 10. The yield strength (YS), ultimate tensile strength (UTS) and elongation (EL) of the Al-6Zn-2Mg-*x*Cu alloys are increased evidently after T6 heat treatment. This is due to a large number of nano-sized GP

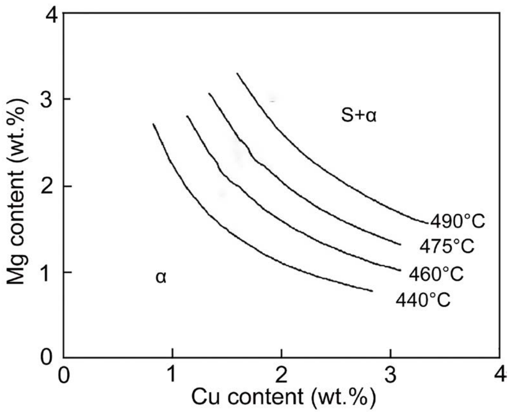


Fig. 7: Solid solubility line of Al_2CuMg phase in Al-6Zn-*y*Mg-*x*Cu alloy with the temperature ranging from 440 °C to 490 °C [13]

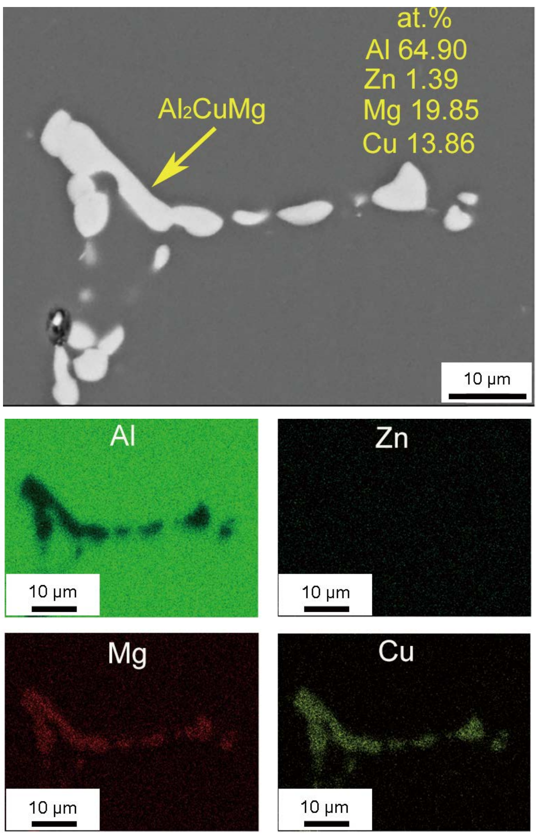


Fig. 8: SEM and EDS of undissolved phase in solution-treated Al-6Zn-2Mg-2Cu alloy

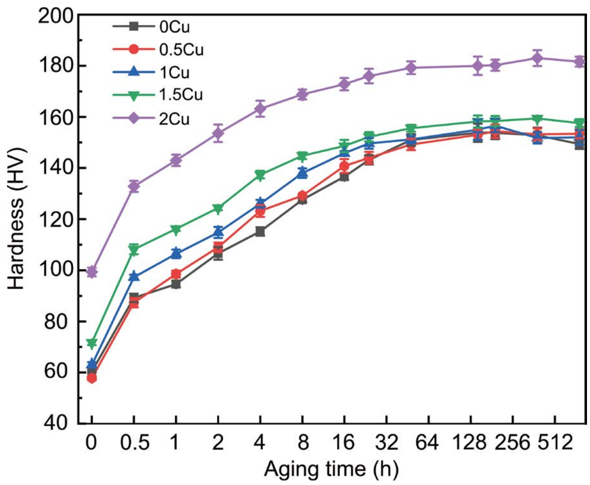
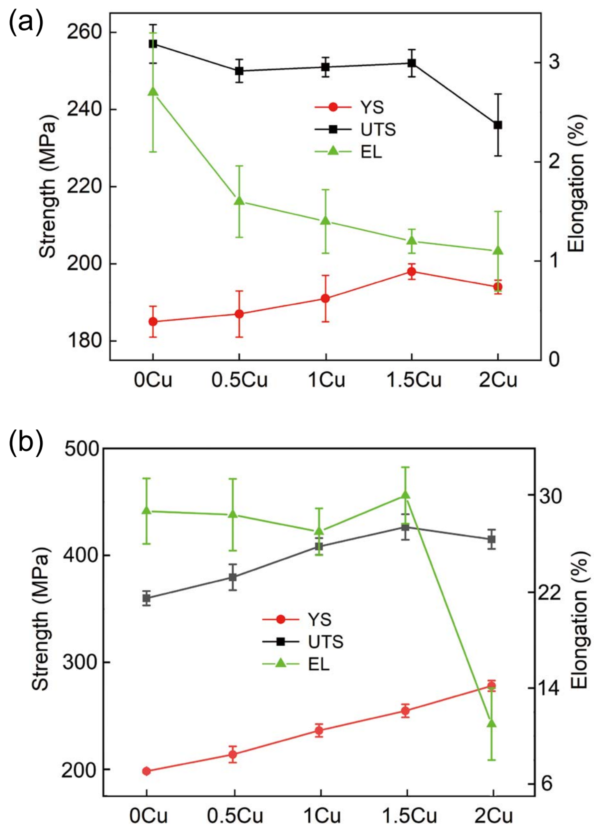


Fig. 9: Dependence of the hardness of solution-treated Al-6Zn-2Mg-*x*Cu alloys on the aging time



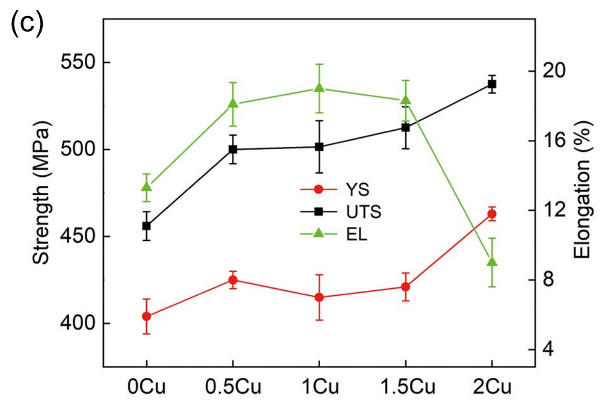


Fig. 10: Mechanical properties of Al-6Zn-2Mg-*x*Cu alloys at the state of: (a) as-cast; (b) solution-treated at 470 °C for 8 h; and (c) solution-treated at 470 °C for 8 h followed by aging at 120 °C for 24 h

zone and η' ($MgZn_2$) phase precipitates in the α-Al matrix, which can effectively hinder the dislocation movement [15]. For the T6 treated alloys, the UTS, YS and EL of 0Cu are about 460 MPa, 400 MPa and 14%, respectively, and the values of 0.5Cu alloy are higher than that of 0Cu alloy and reach about 500 MPa, 420 MPa and 18%, respectively, while the values of 1Cu and 1.5Cu are almost the same with 0.5Cu. Besides, the YS and UTS increase while EL dramatically decreases when the addition of Cu is increased to 2wt.%. The decrease of elongation is related to the stress concentration and micro-cracks caused by Al_2CuMg phase during the tensile test.

4 Conclusions

(1) The high strength Al-6Zn-2Mg-*x*Cu alloys were successfully fabricated by high solid fraction semi-solid rheo-diecasting method. The microstructure of rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys consists of typical globular primary α-Al grains, smaller secondary α-Al grains and eutectic phases, and the average grain size and shape factor of as-cast Al-6Zn-2Mg-*x*Cu alloys is about 80 μm and 0.45, respectively.

(2) The dominant eutectic phases in rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys are η phases. At the as-cast state, the volume fraction of eutectic phases increases with increasing Cu content. After T6 heat treatment, the eutectic phases will dissolve into Al matrix when Cu content is lower than 1.5wt.%, while some eutectic phases transform into S-Al_2CuMg phases and remain at grain boundaries when Cu content reaches 2wt.%.

(3) After T6 heat treatment, the mechanical properties of rheo-diecasting Al-6Zn-2Mg-*x*Cu alloys are significantly enhanced. The ultimate tensile strength of the Al-6Zn-2Mg-*x*Cu alloys is about 500 MPa, the yield strength is about 420 MPa, and the elongation is about 18% when Cu content is between 0.5wt.% to 1.5wt.%. The ultimate tensile strength and yield strength both increase with increasing Cu content, while the elongation evidently decreases when Cu content is increased from 1.5wt.% to 2wt.%.

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