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Solidification and homogenization behaviors of Al-9.1 Zn-2.1 Mg-2.2 Cu-0.1 Zr-0.07 Ce alloy

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Abstract

Solidification and homogenization behaviors of Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy were investigated. Obvious grain refinement was observed in as-cast alloy by Ce modification, the main mechanism of the grain refinement is that the redistribution of solute during solidification leads to the increase of the supercooling degree of the solid/liquid interface front component and the blocking effect of primary Ce enrichent phase. Serious non-equilibrium eutectic phase formed during rapid solidification and a phase transformation from Mg(Zn, Al, Cu)2 to few Al2CuMg were also occurred even with 9.1 wt% Zn content at present alloy, owing to trace Ce as solute can hinder the diffusion of Cu atoms in Mg(Zn, Al, Cu)2 phase effectivley. Obviously the solidification Mg(Zn, Al, Cu)2 phase was eliminated thoroughly and a larger number of disperse Al1Zr was precipitated effectively by a double-stage homogenization treatment. Thus the homogenization at 435 °C for 8 h and then at 470 °C for 32 h is identified as an optimum homogenization treatment in this experimental alloy.

1. Introduction

Owing to higher specific strength, ultra-high strength Al–Zn–Mg–Cu alloys with acceptable toughness might be widely used in military and aerospace industries [1, 2]. Generally the strength of Al–Zn–Mg–Cu alloys are usually increased with the Zn content [3]. However, the main problem in the commercial application of these high Zn content alloys is their serious dendritic segregation and coarse intermetallic phases which would be formed during the rapid solidification processing. Particularly the coarse undissolved phases are detrimental to the mechanical properties of these alloys [4]. Normally the hight alloying alloy usually results in ultra-high strength at the expense of the alloy ductility [5]. An optimized homogenization process is an indispensable step in order to improve the microstructures, mechanical properties, and formability of Al-Zn-Mg ingots [6].

Several intermetallic phases, such as MgZn2, Al2Mg3Zn3, Al2CuMg, Al2Cu, Al2Cu3Fe, Al2Fe5, and Mg2Si might exist in commercial Al–Zn–Mg–Cu alloys below the soliud temperature [7, 8]. The residual bulky intermetallic compounds (> 1 μm) typically first result in brittle fracture during deformation, which may be the source of crack initiation for Al–Zn–Mg–Cu alloys. Thus, these coarse residual phases can damage the toughness, fatigue properties [9], and corrosion properties of Al–Zn–Mg–Cu alloys [10]. Some studies indicated that Al2CuMg phase can be formed in both as-cast and homogenized alloys [11, 12]. However, Liu et al [4] found that the phase transition from Mg(Zn, Al, Cu)2 to Al2CuMg phases was very difficult when Zn content was higher than 8 wt% during homogenization and the main residual phase was AlZnMgCu phase. In addition to considering these intermetallic phases, desired grain size and interparticle distance are important to achieve outstanding properties in the Al–Zn–Mg–Cu alloys [5, 13]. Rare earth elements such as Er, Sc and Ce have been added to improve the performance of Al alloy by refining the as-cast grain size, inhibiting recrystallization and promoting the precipitation of α′ phase during artificial aging [14–16]. Zhang et al [17] added Ce to an Al-18%Si (wt%) alloy and observed that the morphology of the primary Si and eutectic Si could be effectively modified from fibrous to lamellar shape. There have also been many reports on the effect of Ce on the morphology of
intermetallic compounds of 7XXX series aluminium alloys, e.g., a refined dendritic structure with the precipitate morphology changing from spherical to needle shape in 7055Al alloy [18] and a refined eutectic microstructure in Al-6.7Zn-2.6Mg-2.6Cu (wt%) alloy [16]. In addition, nano-sized Al$_3$Zr dispersoid that acted as a recrystallization inhibitor by a grain and sub-grain boundary pinning mechanism was also precipitated during homogenization in the Al–Zn–Mg–Cu–Zr alloys [19]. The effectiveness of Al$_3$Zr dispersoids for stabilizing grain structure mainly depended on their dispersion which were strongly effected by the homogenization scheme [20, 21].

Despite several studies of the as-cast microstructure and homogenization treatment in commercial Al–Zn–Mg–Cu alloy, less attention was focused on the solidification behavior and evolution of non-equilibrium eutectics during homogenization in Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce alloy (wt%) with ultra-high Zn content. Herein, the addition of 0.07 wt% Ce in this alloy can promote the grain refinement obviously, which can partly offset negative effect as plasticity decreases due to high alloying Al–Zn–Mg–Cu alloy, and subsequently optimize the working performance. Thus, the main aim of present work is investigate the solidification behavior and phase evolution during homogenization, and optimize its parameters of a Al-9.1Zn-2.1Mg-2.2Cu-0.1 Zr-0.07Ce (wt%) alloy.

2. Experiments

The experimental alloy with chemical composition of Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) was prepared with purity Al, Zn, Mg, and master alloys as Al-51.5Cu, Al-3.29Zr, Al-10Ce (wt%). The specimens with dimensions of 15 mm × 15 mm × 15 mm were used for homogenization treatment. Figure 1 shows the DSC curve of the as-cast alloy. Only one sharp endothermic peak at 474.1 °C approximately, which is related to the dissolution of non-equilibrium eutectics was observed. In order to avoid overburning during homogenization and considering the accuracy of the furnace, 470 °C is chosen as the homogenization temperature in this work. Single-stage homogenization treatments were performed at 470 °C for 8, 16, 24, 32 and 40 h, respectively. Meanwhile, a double-stage homogenization treatment 435 °C for 8 h and then at 470 °C for 32 h is implemented as a comparison.

The microstructure of the alloys was characterized by optical microscopy (OM), scanning electron microscopy (SEM), and x-ray diffractometer (XRD). In addition, differential scanning calorimetry (DSC) was used to measure the overburnt temperature of non-equilibrium eutectics. The precipitated Al$_3$Zr particle was characterized by transmission electron microscopy (TEM). And local chemical compositions and the distribution of alloy element were determined by electron probe micro-analyzer (EPMA) equipped with wavelength dispersive spectroscopy (WDS). The average radius, area fraction of the Al$_3$Zr dispersoid were quantified by image analysis of digitized TEM bright field images.
3. Results and Discussion

3.1. Microstructure of as-cast alloy

The optical micrographs of the as-cast experimental alloys without and with 0.07 wt% Ce addition are shown in figure 2. Serious dendritic segregation inner grain exists in the as-cast Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr (wt%) alloy, as shown in figure 2(a). While the grain size of the as-cast alloy with 0.07 wt% Ce addition is refined remarkably from about 300 μm to 100 μm. Obvious dendritic segregation is mainly along the grain boundary and is seldom seen in grain interior, as shown in figure 2(b). Reference to the Al-Ce binary phase diagram [22] shows that an eutectic reaction occurs at 650 °C, where a composition of 0.05 wt% Ce, resulting in the formation of α (Al) and the Al₄Ce intermetallic phase. However, the rare earth Ce and Al form eutectic phase, and most of the Al-Ce compounds are not satisfied as the ‘structure matching’ principle of the matrix α (Al) heterostructure, so the rare earth phase containing Ce is difficult to be a heterogeneous nucleation core of the α (Al). The possible reason for the grain refinement is that supersaturated Ce atoms were expelled from the solidified grains and accumulated at the front of the interface between solid and liquid during solidification. When the Ce concentration approaches the eutectic point, primary Ce enrichment phase will form by the decomposing eutectic reaction. The primary Ce enrichment phase will prevent further growth of the grains. The process is similar to the grain refining of Al–Mg–Mn–Zr alloy with Er addition [23]. Therefore, the redistribution of solute during solidification leads to the increase of the supercooling degree of the solid/liquid interface front component and the blocking effect of primary Ce enrichment phase, which are the main mechanism of rare earth element Ce refining the grain structure of this alloy.

According to a substantial difference of brightness and morphology of intermetallic microstructure in as-cast Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy, intermetallic phase can be obviously classified to seven types, mark as phase A, B, C, D, E, F and G, as shown in figure 3. The composition of the intermetallic phases present in figure 3, obtained from quantitative x-ray WDS analysis, is shown in table 1. The main solidification phase with network lamellar eutectic morphology (Phase D) is determined to be the mixture of α (Al) and Mg(Zn, Al, Cu)₂. And the Mg(Zn, Al, Cu)₂ phase is also proved by the edge of the lamellar eutectic phase again (Phase B). A little gray secondary phases mingle with Mg(Zn, Al, Cu)₂ phase (Phases A and G) which rich in Al, Cu are identified as Al₄Cu phase. Very few deep gray rod-like phase (Phase C) contains Cu and Fe is close to stoichiometric Al₄Cu₂Fe phase in chemical composition. In addition, the bright bulk-like particles (Phases E and F) with the atom fraction of Al: Cu: Ce is close to 8:4:1 are Al₄Cu₃Ce phase, which is consistent with the previous work [16, 24].

The segregation of Al, Zn, Mg, Cu, Zr and Ce elements are shown in figure 4. Obviously serious element segregation of Zn, Mg, Cu and Ce occurs along grain boundaries in the as-cast alloy. According to the mapping observation, the segregation degree is Cu > Zn > Mg. The element Ce tends to segregation with Cu which forms CuCe enrichment phase. The concentration of the main segregation elements decreases from grain boundary to inside. Thus, a reasonable homogenization treatment is required to eliminate serious dendritic segregation in the as-cast alloy.

3.2. Microstructure of homogenized alloy

Figure 5 shows the backscattered electron image of a double-stage homogenized alloy. The chemical composition of these residual phases are listed in table 2. Compared with the as-cast alloy, Mg(Zn, Al, Cu)₂ phase disappears and a few isolated Al₄CuMg phase (Phase I) forms after homogenization. It is observed that Mg(Zn, Cu, Al)₂ phase dissolve into matrix or partially transforms into Al₄CuMg phase during homogenization. Besides,
a few Al₈Cu₄Ce (Phase H) phase and very little Al₂Cu₁Fe (Phase J) phase could still be detected in the homogenized microstructure, indicating that these particles have seldom dissolved in α (Al) matrix during homogenization.

Figure 6 shows x-ray diffraction patterns of the alloy in the as-cast and homogenized states. The phases consist of α (Al), MgZn₂, Al₂Cu₁, and Al₈Cu₄Ce in the as-cast alloy. Hence, the lamellar eutectic Mg(Zn, Al, Cu)₂ phase observed in figure 3 should be having the MgZn₂ phase crystal structure. It is inferred that Al and Cu will
To substitute for Zn at the lattice position of Zn in MgZn$_2$ phase with the formation of Mg(Zn, Al, Cu)$_2$ \cite{25}. While Al$_7$Cu$_2$Fe phase cannot be observed by x-ray diffraction, owing to the small amount or size of these phases which are the experimental limit for the x-ray diffractometer used \cite{26}. With the prolonging of homogenization time, MgZn$_2$/Mg(Zn, Al, Cu)$_2$ phase dissolves into matrix gradually until completely dissolved. As reflected in diffraction peak of MgZn$_2$/Mg(Zn, Al, Cu)$_2$ phase disappears absolutely after homogenization at 435 °C/8 h + 470 °C/32 h, which indicates that Mg(Zn, Al, Cu)$_2$ phase dissolves into α (Al) matrix completely. Meanwhile, a weak diffraction peak of Al$_2$CuMg phase appears, which is in accordance with WDS analysis results in homogenized experimental alloy, as shown in figure 5. However, The Al$_8$Cu$_4$Ce phase with high-temperature stability hardly dissolved during homogenization.

Microstructure evolution of the Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy during homogenization is shown in figure 7. The volume fraction of the residual phases declines significantly with the increase of homogenization time from 8 to 40 h. And the residual phases become thinner and the distribution of the phases becomes discontinuous and sparse. Compared with the single-stage homogenization treatment at 470 °C for 40 h, lesser volume fraction of residual phase was observed at a double-stage homogenization treatment. The line scanning analysises of the solidification phase in as-cast alloy and residual phase after a double-stage homogenized alloy are shown in figure 8. Obviously, the distribution of Zn, Mg and Cu elements along interdendritic region varied periodically in as-cast alloy. The grain boundary phases are almost eliminated and few residual phases could be observed after a double-stage homogenization treatment. The element Cu and Mg segregation together on the Al$_2$CuMg phase, where elements Zn is almost eliminated after homogenization.

### 3.3. Microstructure evolution analysis during homogenization

Generally, the typical solidification phases in the Al–Zn–Mg–Cu alloy are Mg(Zn, Al, Cu)$_2$, small amounts of Fe-rich phase and Al$_2$Cu phases may also be observed \cite{11, 27}. Network lamellar eutectic phases dissolved into matrix gradually, and large amounts of deep gray Al$_2$CuMg phase formed after homogenization, as shown in figure 5 (Phase I). It is well known that the diffuse velocity of Cu is lower than that of Zn and Mg, which results in an accumulation of Cu in these regions during homogenization. The growth rate of Al$_2$CuMg phase is mainly controlled by the diffusion of Cu and Zn between the Mg(Zn, Cu, Al)$_2$ phase and α (Al) matrix \cite{23}. Although Liu et al \cite{4} indicated that the phase transition from Mg(Zn, Al, Cu)$_2$ to Al$_2$CuMg phases was very difficult during homogenization when Zn content was higher than 8 wt%, while obvious transition from Mg(Zn, Al, Cu)$_2$ to Al$_2$CuMg phases was observed at present alloy even with 9.1 wt%Zn content. It can be inferred that the particular introduction of Ce in present Al-9.1Zn-2.1Mg-2.2 Cu-0.1Zr alloy promotes the

![Figure 5. Backscattered electron images of homogenized Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy at 435 °C/8 h + 470 °C/32 h.](image)

| Table 2. Chemical composition of second phase in as-homogenized alloy measured by WDS (at%). |
| --- |
| Phase | Al | Zn | Mg | Cu | Zr | Fe | Ce | Phase |
| H | 56.8445 | 8.7635 | 1.2425 | 25.1083 | — | 1.4422 | 6.5830 | Al$_9$Cu$_4$Ce |
| I | 51.1837 | 1.2160 | 25.6502 | 21.9270 | — | — | 0.0230 | Al$_5$CuMg |
| J | 73.3450 | 1.7283 | 0.5311 | 10.6915 | — | 13.5675 | 0.0601 | Al$_2$Cu$_4$Fe |

Figure 5. Backscattered electron images of homogenized Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy at 435 °C/8 h + 470 °C/32 h.
Figure 6. XRD patterns of Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy. (a) as-cast; (b) homogenized.

Figure 7. Backscattered electron micrograph of Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy homogenized under different conditions: (a) 470 °C/8 h; (b) 470 °C/16 h; (c) 470 °C/24 h; (d) 470 °C/32 h; (e) 470 °C/40 h; (f) 435 °C/8 h + 470 °C/32 h.
formation of Al$_2$CuMg in the homogenization process, owing to even trace Ce as solute can hinder the diffusion of Cu atoms in Mg(Zn, Al, Cu)$_2$ phase effectively [28].

The TEM bright field images of the Al$_3$Zr dispersoids after both single-stage and double-stage homogenization treatments and the corresponding selected area diffraction (SAD) pattern are presented in figure 9. Compared with the single-stage homogenization, a higher number density of Al$_3$Zr dispersoids are obtained for the double-stage homogenization. The average radius ($r$) and area fraction of the Al$_3$Zr dispersoids ($f$) are listed in table 3. It is obvious that the double-stage homogenization with a first step held at 435 °C for 8 h has a significant effect on the precipitation of Al$_3$Zr dispersoids. The concrete forms of that is resulting in a denser and finer distribution of the dispersoids which means the $f/r$ is relatively higher in double-stage than in single homogenization treatment. The value of $f/r$ is increased from 89.08 to 123.78 ($\mu$m$^{-1}$) due to additional low temperature heat treatment. The reason can be traced back to the main factor influencing the dispersoids precipitation during homogenization, namely, the supersaturation of the solid solution which is act as the driving force for nucleation. That is the pre-homogenization under a lower temperature procedure promotes the Al$_3$Zr dispersoids nucleation for developing an optimized distribution. The double-stage homogenization treatment can result in a more uniform Al$_3$Zr dispersoids distribution than that achieved by single-stage homogenization treatment, owing to the first stage at a relatively low temperature can be used to promote the nucleation of dispersoids and followed by a further higher temperature can optimize the growth and coarsening of the dispersoids effectively [19].

![Figure 8](image-url)
4. Conclusions

Herein, solidification and homogenization behavior of the Al-9.1Zn-2.1Mg-2.2Cu-0.1Zr-0.07Ce (wt%) alloy were studied. Obviously the addition of 0.07 wt% Ce results in grain refinement remarkably. The solidification phase Mg(Zn, Cu, Al)\textsubscript{2} dissolves into \( \alpha \) (Al) matrix and transform to Al\textsubscript{2}CuMg phase simultaneously during homogenization even with 9.1 wt% Zn content at present alloy. It can be inferred that the particular introduction of Ce promotes the formation of Al\textsubscript{2}CuMg during homogenization, owing to even trace Ce as solute can effectively hinder the diffusion of Cu atoms. A double-stage homogenization treatment at 435 °C for 8 h and then 470 °C for 32 h is identified as an optimum homogenization treatment of the alloy, due to the Mg(Zn, Al, Cu)\textsubscript{2} phase is eliminated thoroughly and a larger number of Al\textsubscript{3}Zr dispersoids are precipitate.

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