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High-pressure homogenization treatment of Al–Zn–Mg–Cu aluminum alloy

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Absract The microstructures and aging hardening response of Al–12Zn–3.5Mg–3.0Cu–0.14Zr aluminum alloy after a high-pressure homogenization treatment at 750 °C for 45 min under 5 GPa were investigated. The results showed that the constituent phases dissolved completely and formed α-Al single-phase solid solution comparable to that formed after ambient-pressure homogenization at 450 °C/96 h + 460 °C/128 h. The complete dissolution of the constituent phases increased the solubility of the alloying elements, as well with the over-burning temperature and aging hardness.

Introduction

It is well-known that the strength of the 7000 series aluminum alloys is dependent on the concentration of Zn and Mg within the matrix, which result in the formation of fine nanometer scale precipitates during aging, such as GP zones, η' meta-stable phases and η equilibrium phases [1]. Cu is incorporated into these precipitates, and increases the stress corrosion resistance and strength of the alloys. In order to further increase the strength of the 7000 series aluminum alloys, the contents of Zn, Mg, and Cu need to be increased, which, in turn, lead to a higher fraction of non-equilibrium constituent phases during chill casting, such as T-$Al_{32}(Mg,Zn)_{49}$, η-$MgZn_2$ and S–Al_2CuMg [2–4]. These constituent phases can also contain other elements and form AlZnMgCu quaternary phases. If these constituent phases are not able to dissolve during the subsequent homogenization and solution treatments, the damage-tolerant properties and corrosion resistance of the alloys degrade significantly [5, 6].

Non-equilibrium constituent phases formed during direct chill casting can be decreased during homogenization treatments. However, in order to prevent over-burning of the ingots, homogenization treatments are often carried out below the lowest solidus temperature of non-equilibrium constituent phases, which makes it difficult to completely dissolve the constituent phases.

Under high pressure, the melting points and phase transformation of some metals and alloys change significantly. Zhang et al. [7] reported that the melting point of pure aluminum increased from 665 °C to 1030 °C under a high pressure of 5 GPa. The investigation of the solidification process of some binary aluminum alloys under high pressure showed that the range of the α-Al single-phase was extended compared to that observed in ambient-pressure equilibrium phase diagrams [8].

Therefore, if homogenization treatments of the 7000 series aluminum alloy were carried out under high pressure, the constituent phases might be dissolved completely and rapidly due to the use of a higher homogenization temperature and the broadening of α-Al single-phase field.

Experimental procedures

The materials used for the current study were 7000 series aluminum ingots. The nominal composition of the ingots was Al–12Zn–3.5Mg–3.0Cu–0.14Zr (wt%). The ingots

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were machined into cylindrical rods and homogenized on the cubic-anvil type apparatus at 750 °C for 45 min under a pressure of 5 GPa. Ambient-pressure homogenization of the ingots at 450 °C/96 h + 460 °C/128 h was performed for comparison. After the homogenization treatment, the specimens were quenched into ice water.

The SEM investigations were conducted on a JSM-6360LA scanning electron microscope operating at 20 kV. Phase identification was determined by X-ray diffraction (XRD) with CuKα_1 radiation on D/max 2500PC diffractometer. Differential scanning calorimetry (DSC) was performed using a Q600-TGA/DSC at a heating rate of 10 °C/min to measure the melting points of the constituent phases.

After ambient-pressure homogenization and further solution treating at 470 °C for 1 h, or high-pressure homogenization, both sets of specimens were artificially aged at 120 °C. Vickers hardness testing was carried out on HXD-1000TMC micro-hardness tester.

Experimental results

Typical as-cast microstructures of the ingots are presented in Fig. 1. A large number of non-equilibrium constituent phases are present along primary α-Al grain boundaries, as shown in Fig. 1a. XRD patterns show that these constituent compounds are mostly η phase (Fig. 1b). In the 7000 series aluminum alloys, η phase forms during chill casting when the ratio of Zn to Mg is above 2.2 [9]. In this study, the ratio of Zn to Mg is about 3.4; therefore, η constituent phases dominated in the as-cast microstructures.

Figure 2a shows that a large amount of second-phase particles remain within the matrix even after the ambient-pressure homogenization treatment at 450 °C/96 h + 460 °C/128 h. These residual particles exhibit a block-like morphology after ambient-pressure homogenization which differs from the lamellar configuration in the as-cast microstructures. The presence of the block-like particles indicates that these particles coarsen and agglomerate during ambient-pressure homogenization and unable to dissolve into the matrix at the homogenization temperature. The XRD patterns show that these particles are predominantly η phase accompanied by small amounts of newly formed T phase, as shown in Fig. 2b.

The homogenized microstructures of the ingots under a high pressure of 5 GPa at 750 °Cfor 45 min are shown in Fig. 3a. The constituent phases dissolved completely as compared with the alloy that received the ambient-pressure homogenization treatment. The XRD results showed that only diffraction peaks of α-Al solid solution were observed, indicating that the constituent phases dissolved completely, as shown in Fig. 3b.

It is further noted that the duration of the high-pressure homogenization treatment was also shortened significantly as compared with that of the ambient-pressure homogenization treatment, which has great significance in commercial applications.

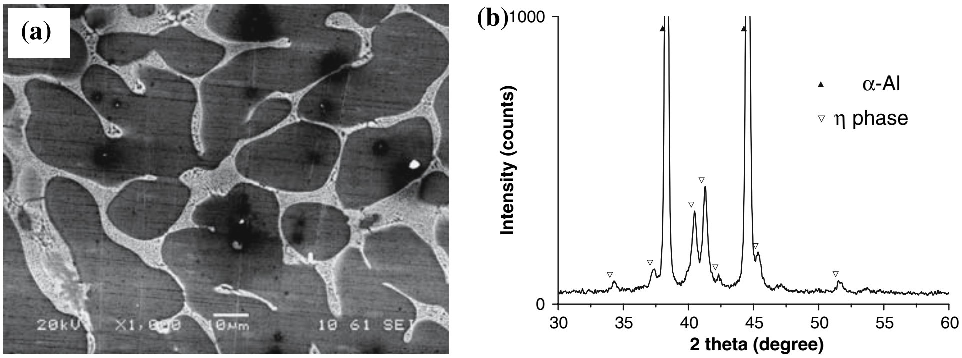


Fig. 1 As-cast microstructures: (**a**) SEM image, (**b**) XRD pattern

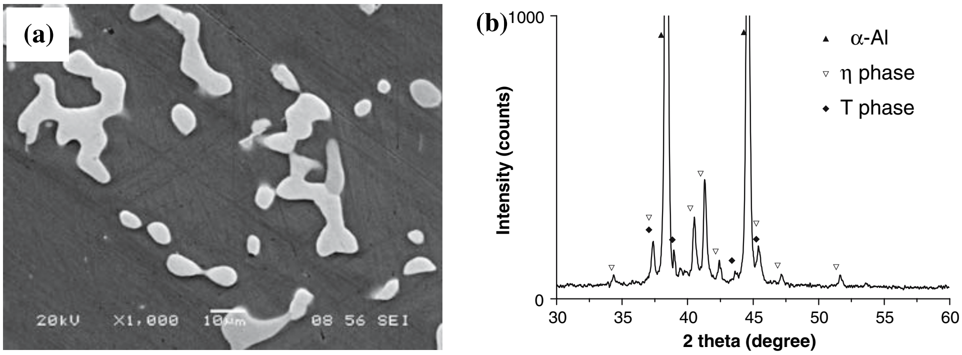
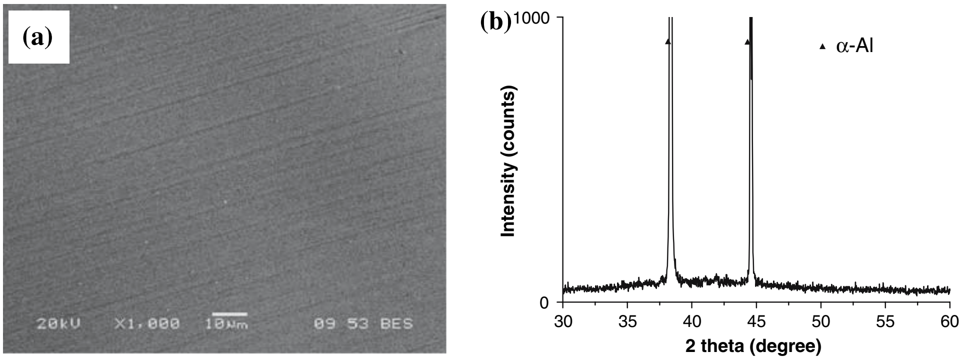


Fig. 2 Microstructures of the samples after ambient-pressure homogenization at 450 °C/96 h + 460 °C/128 h: (**a**) SEM image, (**b**) XRD pattern

Fig. 3 SEM microstructures of the samples after high-pressure homogenization treatment at 750 °C: (**a**) SEM image, (**b**) XRD pattern



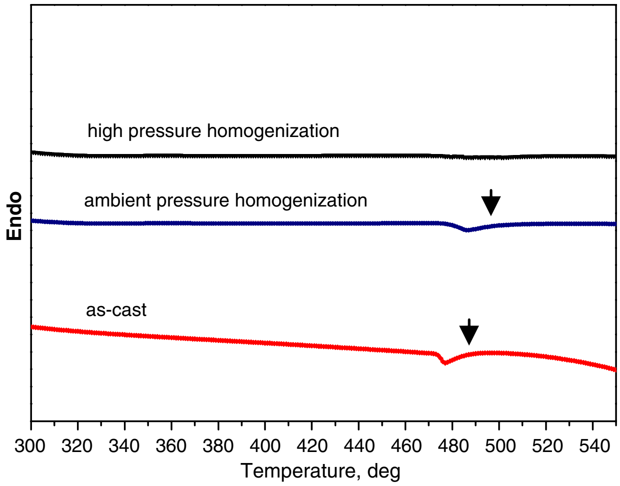


Fig. 4 DSC thermograms of the ingots

The results of the DSC analyses of the three sets of specimens—(1) as-cast, (2) homogenized under ambient pressure at 450 °C/96 h + 460 °C/128 h, and (3) homogenized under high pressure of 5 GPa at 750 °C for 45 min—are shown in Fig. 4. The endothermic peaks caused by the dissolution of the non-equilibrium constituent phases were not present for the ingot treated under high-pressure homogenization, which is correlated with the elimination of the non-equilibrium constituent particles.

The hardening curves for the specimens aged at 120 °C after either ambient-pressure solution treatment at 470 °C for 1 h and or high-pressure homogenization are shown in Fig. 5. The alloy homogenized under high-pressure exhibited higher Vickers hardness than the alloy which had received the ambient-pressure homogenization and solution treatment.

Discussion

The dependence of the melting points of metal and alloys on applied pressure can be calculated according to the Clausius–Clapeyron equation:

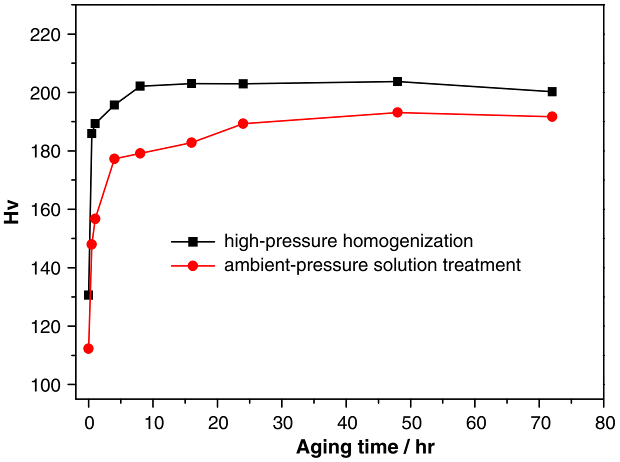


Fig. 5 Hardening curves of the alloys that had previously been solution-treated under ambient-pressure or homogenized under high-pressure, respectively

$$\frac{dT_m}{dP} = \frac{T_m(V_2 - V_1)}{\Delta H_m} \tag{1}$$

where T_m and ΔH_m are the melting point and latent heat of fusion of the alloy under ambient pressure, respectively. V_1 and V_2 are the volumes of solid and liquid phases of 1 kg, respectively. According to the relationship, the melting point of pure aluminum increases from 665 °C to 1030 °C under high pressure of 5 GPa [7]. In our experiments, the alloys do not melt during the high-pressure homogenization at 750 °C, which indicates that the solidus line of α-Al single-phase field is increased and its range is extended. Therefore, higher homogenization temperature can be used to homogenize the ingots.

Comparing Figs. 2b and 3b, the presence of the residual η phase and newly formed T phase after ambient-pressure homogenization signified that the alloy stayed within α-Al + η + T ternary phase field. However, only α-Al single-phase was observed after high-pressure homogenization, which indicated the alloy was in α-Al single-phase field, and η and T phase are not stable at 750 °C under 5 GPa.

For most metals and alloys, the increased pressure is adverse to atomic diffusion. However, in our experiments, the high-pressure homogenization duration is shortened significantly compared with that of ambient-pressure homogenization. The results were due to two factors. Firstly, under 5 GPa, the solidus of the alloy increased; therefore, a higher homogenization temperature could be used to treat the ingots. The use of a higher homogenization temperature will accelerate atomic diffusion. On the other hand, the alloy lies in α-Al single-phase field at 750 °C under 5 GPa, which decrease the stability of η constituent phases during high-pressure homogenization.

The complete dissolving of the constituent phases results in the increase of solid solubility in the matrix, therefore, the lattice distortion in α-Al solid solution and the amount of precipitates during consequent artificial aging increase, which cause higher aging hardness than that after ambient-pressure solution treatment, as shown in Fig. 5.

Conclusions

Two conclusions can be drawn from the above results:

(1) The constituent phases are dissolved rapidly and completely by high-pressure homogenization, which increase the solubility of the alloying elements in the matrix. Therefore, the over-burning temperature and age-hardening response increase.

(2) The Al–12Zn–3.5Mg–3.0Cu–0.14Zr aluminum alloy lies in the α-Al single-phase field at 750 °C under 5 GPa.

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