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Influence of quench-induced precipitation on aging behavior of Al–Zn–Mg–Cu alloy

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Abstract: The effects of quenching and aging (T6, T7 and RRA) on the microstructural evolution of an Al–Zn–Mg–Cu alloy were investigated by hardness test, optical microscopy (OM), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) measurements. It is found that the hardness of T6 aged sample after water-quenching is the highest. The quench sensitivities of T7 and RRA are almost the same, which are 1.2% higher than that of T6. TEM observation shows that the quench sensitivity for the studied alloy is mainly caused by heterogeneous precipitation during slow quenching. Many η phases precipitate on Al₃Zr dispersoids inside recrystallized grains and at (sub) grain boundaries, while T and S phases form in the substructure with high density of dislocations and defects. After aging, the η' precipitates are coarser in the vicinity of equilibrium η phase. However, the size and morphology of the precipitates show different characteristics among T6, T7 and RRA treatments. The DSC results are highly consistent with the TEM observation. The DSC curves of T6 aged samples are different from those of T7 and RRA aged samples, which also reflects the differences on the microstructure.

Key words: Al-Zn-Mg-Cu alloy; quenching; quench sensitivity; aging; precipitation

1 Introduction

Al–Zn–Mg–Cu alloys have a variety of applications in automotive and aircraft industries due to their high age-hardening response and low density [1,2]. The precipitation evolution of Al–Zn–Mg–Cu alloys during aging has been thoroughly studied [3,4], and the precipitation sequence usually represents as [5]:

solid solution \rightarrow coherent GP zone \rightarrow semi-coherent η' \rightarrow incoherent η (MgZn₂)

Instead of η phase, GP zone and η' phase are proved to be responsible for the peak strength of Al–Zn–Mg–Cu alloys. PHILIP and RICHARD [6] observed that 7050 alloy with maximum strength had a microstructure of an approximately equivalent GP zone and η' phase concentration.

It is well known that alloys aged by T6 temper could bring about a maximum strength but low stress corrosion cracking (SCC) resistance [7]. T7X treatments of two-stage isothermal aging are invented to improve the SCC resistance but lead to various degree of decrease in strength [8]. Retrogression and re-aging (RRA) treated alloys have good strength and corrosion resistance. 7055–T77 aluminum alloy provides yield strength 10% higher than that of 7150–T6 aluminum alloy and about 30% higher than 7075–T76 aluminum alloy [9].

DSC could be used to study the precipitations existing before the scan, and the following dissolution or transformation reactions [10]. BUHA et al [11] reported that during the DSC run of aluminum alloy 7050 after there were five exothermic reactions solution, corresponding to five kinds of precipitations observed, GP zone, η' , η , T and S. For the alloys after aging, LIM et al [12] investigated the effect of Zn/Mg ratio on the quench sensitivity of aluminum alloy 7175 thick forging by means of DSC. KAMP et al [13] investigated aluminum alloy 7449 and found various aging treatments could change the DSC curves in a complex manner. However, in previous study on quench sensitivity, less attention has been paid to the comparison of different aging behaviors by DSC.

It is well known that the aging precipitation process

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of Al-Zn-Mg-Cu alloy is quite sensitive to quenching condition. Especially for large-scale products, the cooling rate is different between the surface and the center, thus resulting in heterogeneity and drop in properties after ageing. According to previous work [14–16], there are many equilibrium η phase particles precipitated on Al₃Zr particles inside recrystallized grains and at grain boundaries during slow quenching. Investigations of aging treatments are mostly limited to the fast guenched samples, but the interaction of guenchinduced phase and aging precipitation has not been mentioned in the previous studies. In this work, the effect of quench-induced precipitation on the aging behavior was investigated in order to clarify the main reasons for quench sensitivity and elucidate the differences of precipitation after different aging treatments.

2 Experimental

Experiments were carried out on a hot-rolled Al-Zn-Mg-Cu alloy sheet with a chemical composition of 8.02% Zn, 1.75% Mg, 1.99% Cu and 0.15% Zr (mass fraction). Rolled sheet with 2.5 mm thickness was solution heat treated at 470 °C for 1 h, then the samples were quenched into water and air at room temperature, respectively. Extremely high and low quenching rates were obtained. The average quenching rates were about 500 and 2 °C/s measured by thermal couples, respectively. After quenching, the samples were subjected to three different aging treatments immediately, named T6 (aging at 120 °C for 24 h), T7 (aging at 120 °C for 2 h followed by a linear heating at 160 °C for 10 h) and RRA (aging at 120 °C for 24 h followed by heating at 180 °C for 30 min, and then re-aging at 120 °C for 24 h).

Vickers hardness tests of the aged samples were carried out on a Model HV–10B instrument under a load of 29.4 N. Grain structure was observed by Olympus PMG3 optical microscopy (OM). Microstructure observations were performed on a TECNAI G²20 microscopy operating at 200 kV. Samples for TEM observation were prepared by mechanical polishing to 100 μ m, punching into discs with diameter of 3 mm, and double-jet electro-polishing in a liquor of 20% nitric acid and 80% methanol operating at –30 °C and 15 V. Calorimetric measurements were made using NETZSCH STA 200F3C DSC instrument. The sample discs with diameter of 5 mm and mass of about 15 mg were heated from 30 to 470 °C at a heating rate of 10 °C/min.

3 Results

3.1 Age hardening

Figure 1 shows the age hardening results of the

alloy with different heat treatments. The aging behavior of samples aged at 120 °C, which was quenched in water or in air, is shown in Fig. 1(a). Relative hardness is used to evaluate the quench sensitivity, the value of which is the ratio of the absolute hardness to the hardness of the water-quenched sample. It is clearly shown that the hardness of the water-quenched sample increases more rapidly than the air-quenched one, and the quench sensitivity becomes relatively low after aging for 20 h. As shown in Fig. 1(b), after T6 heat treatment, the hardness reaches the peak value, and the hardness of the water- and air-quenched samples were HV195.7 and 146.5, respectively. Compared with T6 treated sample, the hardness of T7 and RRA aged samples decrease by about 4.2% and 2.9% for the water-quenched samples, respectively. While for air-quenched samples, the hardness of T7 and RRA aged samples decrease by 5.7% and 5.3%, respectively. The quench sensitivity of samples through RRA and T7 was almost the same, which is about 1.2% higher than that of T6.



Fig. 1 Hardness of samples aged at 120 $^{\circ}$ C (a) and by T6, RRA and T7 heat treatments (b)

3.2 Optical microstructure

Figure 2 shows the optical micrographs of the studied alloy after solution heat treatment. The grain consists of a partially recrystallized structure (bright

zones), and the fraction of recrystallization is about 20%. The recrystallized grain size in the short transverse direction is about 15 μ m. It is evident that subgrain structure (dark zones) is the main structure of the studied alloy.



Fig. 2 Optical micrographs of grain structure after solution heat treatment (L—Longitudinal direction; S—Shore transverse direction; T—Long transverse direction)

3.3 Quench-induced precipitation

In order to investigate the heterogeneous precipita-

tion induced by quenching, the microstructure of the solution heat treated alloys was observed by TEM, as shown in Fig. 3. When quenched in water, quench-induced precipitation is suppressed and only Al_3Zr particles could be seen inside the grains (Fig. 3(a)).

Air quench leads to heterogeneous precipitation. It is observed that coarse equilibrium η phases mostly nucleate on the Al₃Zr particles inside the recrystallized grains and at the grain boundaries (Fig. 3(b)). The Al₃Zr particles in recrystallized grains could provide effective nucleation sites for η phases because they become incoherent with Al matrix after cutting through by grain boundaries during recrystallization [17]. The η phases are in two shapes, plate-like phase with most hexagonal shape in size of about 200 nm, and long laths phase with an average size of about 200 nm×25 nm. Some parallel needle-like phases could be observed in the substructure (Fig. 3(c)), which are thought to be S (Al₂CuMg) phases according to BUHA et al [11] and GODARD et al [18]. Particles with blocky morphology (most irregular round and hexagonal) are found in the subgrain region with high density of dislocations, as shown in Fig. 3(d), those particles have a diameter



Fig. 3 TEM micrographs of studied alloy after solution heat treatment and quenching: (a) No quench-induced precipitate after water quenching; (b) Equilibrium η precipitates in recrystallized structure, (c) *S* phase in substructure after air quenching; (d) *T* phase in substructure after air quenching

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ranging from 200 to 350 nm, and are approved to be T (Al₃Zn₃Mg₃ or (AlZn)₄₉Mg₃₂) phases, which will be discussed in the next section.

Precipitation of η , T and S phases during air quenching leads to fewer solute atoms in the supersaturated solid solution, thus lowers the aging hardening effect of air-quenched alloys, which is demonstrated in Fig. 1.

3.4 Influence of quench-induced precipitation on aging behavior

3.4.1 Precipitation after T6 heat treatment

TEM observation was carried out on the T6 aged alloys and quenched samples, as shown in Fig. 4. In the





case of water quenching, Figure 4(a) shows that very fine GP zones and η' phase are distributed homogeneously inside the recrystallized grains, coarse and less spaced η phase is distributed at grain boundaries, and no obvious precipitate free zone (PFZ) is observed. It is known from Fig. 4(b) that precipitates inside subgrains are also very fine GP zones and η' phase, and η' phase is a little coarser in the region rich in dislocations.

The microstructure is quite different in the air-quenched alloy from that water-quenched alloy. Precipitates in the air-quenched sample are a little coarser than those in the water-quenched one, as shown in the recrystallized structure of Fig. 4(c). There are many coarser η phase particles at the grain boundaries and the PFZs at different positions are different. The PFZ at the position around the quench-induced η phase has a width of 150 nm, while the PFZ width is about 60 nm near the age-induced η phase. Another phenomenon is that the hardening precipitates adjacent to equilibrium η phases are coarser and sparser than those far away, which could be judged from Fig. 4(d). The precipitates at the former position grow to needle-like η phases with an average size of about 15 nm. While away from the

equilibrium phase, there are a lot of fine η' phase particles in the matrix according to the inset selected area diffraction (SAD) pattern in Fig. 4(d). Figure 4(e) shows that particles with irregular morphology are found in subgrain region rich in dislocations, which also appear in Fig. 3(d). Figure 4(f) shows the SAD pattern from Fig. 4(e) in [111], in which the irregular particles are *T* phase [11] as indicated by arrows in Fig. 4(g), and a high magnification image is shown in Fig. 4(f). 3.4.2 Precipitation after T7 heat treatment

Figure 5 shows the typical microstructures in the alloys after water or air quenching and T7 heat treatment. In the case of water quenching, Figs. 5(a) and (b) show the recrystallized structure and substructure, respectively. Both inside recrystallized grains and subgrains, fine η' phases are homogeneously dispersed, and also some rod-shaped coarse η phases are apparently distributed in the matrix. Precipitates at recrystallized grain boundaries are almost completely discontinuous, and grow to an elliptical shape, with length of about 60 nm. Along the subgrain boundaries, rod-like and lath shaped η phases with size of 15–25 nm are observed, but no evident PFZ is present.



Fig. 5 TEM micrographs of studied alloy after T7 heat treatment: (a) Precipitates in recrystallized structure for water-quenched sample; (b) Substructure for water-quenched sample; (c) Recrystallized grain for air-quenched sample; (d) Subgrain for air-quenched sample

Compared with the water-quenched sample, the matrix precipitates in air-quenched one in Figs. 5(c) and (d) (recrystallized grain and subgrain) are coarser. The aging precipitates grow to rod-shaped and plate-shaped η' or η phases with an average size of (10–20) nm×3 nm, and the precipitates adjacent to quench-induced equilibrium η phase are 1.5–2 times coarser.

3.4.3 Precipitation after RRA heat treatment

Figure 6 shows the microstructures of the studied alloys after water or air quenching and RRA heat



Fig. 6 TEM micrographs of studied alloy after RRA heat treatment: (a) Precipitates in recrystallized grain structure of water-quenched sample; (b) Substructure of water-quenched sample; (c) Substructure of air-quenched sample

treatment. For the water-quenched sample, GP zones and fine η' precipitates are distributed inside recrystallized grains, as shown in Fig. 6(a). At the recrystallized grain boundaries, the precipitates are a little discontinuous and the width of the PFZ is about 50 nm. Some coarser needle-like η phases with size of 10–15 nm are also observed inside the subgrains and at the subgrain boundaries in Fig. 6(b).

The substructure of air-quenched sample is shown in Fig. 6(c). Precipitates inside the subgrains are dense η' and rod-shaped η phases with size of about 15 nm, which are coarser than those of the water-quenched sample. The quench induced η phases along the subgrain boundary have a length ranging from 100 to 200 nm with an apparent PFZ.

3.5 Precipitation reactions in aged alloy

The DSC curves of T6, RRA and T7 aged alloys are presented in Fig. 7. For convenient analysis, DSC curves in Fig. 7 are divided into three regions by the zero heat flow line. The evolution of peaks is consistent with the earlier work by other researchers [6,11,19]. For both water-quenched (Fig. 7(a)) and air-quenched (Fig. 7(b)) alloys, it could be observed that a peak marked A firstly appears in endothermic region I, corresponding to the



Fig. 7 DSC curves of T6, RRA and T7 aged alloys: (a) Water quenched; (b) Air quenched

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reversion of GP zones and fine η' phases. In region II, an exothermic peak marked B highly overlaps with peak A in T6 treated sample, relating to the transformation of GP zones and η' to η phase, which does not appear in RRA and T7 aged alloys. With increasing heating temperature, peaks C and D are attributed to the formation of *T* phase, and subsequently *S* phase, respectively. The higher temperature endothermic reaction of region III (peak E) corresponding to the dissolution of the precipitates present is mostly the formation of η phase.

Differences of the curves between water- and airquenched samples after T6, RRA and T7 aging could be found with the help of the data from Table 1, which will be discussed together with TEM observations in the next section. In the table, T_P denotes the peak dissolution temperature, at which the dissolution reaches the maximum rate.

Table 1 DSC results of kinetic parameters from Fig. 7

Aging condition	Quench medium	$T_{\rm P}$ of peak A/ °C	$T_{\rm P}$ of peaks B, C and D/°C
Т6	Water	179.0	210.7, 240.2 and 265.0
	Air	191.0	217.9, 245.8 and 269.1
RRA	Water	188.4	None, 236.2 and 261.0
	Air	202.1	None, 242.4 and 264.7
Τ7	Water	194.8	None, 236.4 and 261.0
	Air	203.8	None, 242.4 and 264.7

4 Discussion

The microstructure observation shows that about 80% of the structure is substructure of the studied alloy. Discussions are divided into two parts, fast quench and slow quench, to present how quench-induced precipitation and aging conditions influence the hardening precipitates.

4.1 Influence of aging on precipitation of fast quenched Al-Zn-Mg-Cu alloy

The TEM results show that the main precipitates of T6 are GP zones and η' phase. The DSC curve of T6 is apparently different from that of T7 and RRA. First, the T_P of peak A in T6 is 179.0 °C, which is 9.4 °C lower than that of RRA and 15.8 °C lower than that of T7, because most precipitates dissolved in peak A of T6 are GP zones. Second, the peaks A and B of T6 overlap between about 179 and 210.7 °C, which do not appear in T7 and RRA. There are enough fine GP zones and η' phase to transform into η phase in T6, which is consistent with TEM results. Third, the enthalpy (area) of region II in T6 is the highest. It is assumed that there are also extra solute atoms after T6 aging. As the exothermic reactions

of region II are processes of consuming Zn and Mg solute atoms available in the matrix, the enthalpy of which mainly depends on the amount of Zn and Mg remained.

The main precipitates in T7 are mostly η' phases and some η phases. The linear heating process of T7 may bring about a composition segregation effect, so the nucleation of η' phases is asymmetric, part of η' phases could grow to a large extent, which bring about a notable decrease in hardness (Fig. 1(b)). During the DSC run, the T_P of peak A in T7 sample is the highest at 194.8 °C due to the main dissolved phase η' . Few fine precipitates could transform to η phases to form peak B in T7, and the exothermic reactions in region II weaken as few solute atoms are available.

The main precipitates of RRA include GP zones, η' phase and some η phase. The RRA brings about 2.9% decrease in hardness compared to T6. In the present study, most fine GP zones and η' phase dissolute into the Al matrix after retrogression at 180 °C, but with high enough solute concentrations of the studied alloy, equilibrium phases pre-formed at grain boundaries and some relative larger η' phases in the substructure would grow or keep balance rather than disappear. When re-aging, those residual precipitates would grow to η phases, consuming part of the solute atoms, therefore, the hardness effect decreases. During the DSC run, the $T_{\rm P}$ of peak A in RRA is 6.4 °C lower than that of T7, which ascribes to more GP zones in RRA. Compared to T6, RRA treated sample is sufficiently precipitated and some η phases are obtained, therefore, few extra solute atoms are obtained in the matrix, peak B disappears and the enthalpy of region II is relatively low.

4.2 Influence of aging on precipitation of slow quenched Al-Zn-Mg-Cu alloy

For Al–Zn–Mg–Cu alloys, with high concentration of the alloying elements, many equilibrium phases precipitate during slow quenching (Figs. 3(b), (c) and (d)). There are many coarse η phase particles precipitated on Al₃Zr particles inside recrystallized grains and at (sub)grain boundaries, also *T* and *S* phases form in the substructure with high density dislocations and defects, which leads to the loss of average supersaturation and annihilation of vacancies [20]. Therefore, the volume fraction of GP zones decreases after aging, which lowers the nucleation sites for η' phase [18], sequentially causes an evident decrease in age hardening responses (Fig. 1) and increase in the quench sensitivity of the alloys.

Then, after aging, quench-induced η phases make the adjacent aging precipitates coarser and sparser than those far away. In the matrix away from equilibrium phase, as the solute atoms and vacancy concentration are relatively sufficient and homogeneous, precipitates are fine and dense. While due to the depletion of solutes atoms and vacancies in the vicinity of equilibrium phase, nucleation is in competitive mode when aging, and once a nucleus is formed over the energy barrier, it will absorb solute atoms around and grow to a larger extent (especially when aging at T7 and RRA), which further lowers the property and increases the quench sensitivity of the studied alloy. Moreover, as the main structure of the studied alloy is substructure, where precipitation is easy to happen for rich dislocations, the size of the precipitates is relatively bigger. When aging at high temperature for a long time, precipitates in substructure would grow further, thus the quench sensitivity of T7 and RRA is relatively higher that that of T6.

The DSC results are highly consistent with the TEM observation. Quenching rate does not change the peak number and the general trend of the whole DSC curves during the same aging (Fig. 7). But the T_P of peak A is 9-14 °C higher in air-quenched samples, which indicates that fine-scale GP zone and η' phase are not obtained due to the loss of solutes and vacancies concentration during air quenching, and then the amount of fine precipitates is highly diminished. Besides, the exothermic reactions of region II are weaker in air-quenched samples, especially for RRA and T7 (Fig. 7). First, the amount of precipitates formed in region II is mainly associated with the amount of Zn and Mg atoms dissolved in the matrix. Under the same aging condition, quenching in air consumes part of Zn and Mg solute atoms due to the quench-induced precipitation, therefore reduces the total heat effect. Second, with increasing aging temperature and time (RRA and T7), η' phases have already precipitated adequately and grown to a large degree or transformed to η phases (Fig. 6), thus the exothermic reactions would be weaken.

5 Conclusions

1) During slow quenching, many η equilibrium phases precipitate on Al₃Zr particles inside recrystallized grains and at (sub)grain boundaries, while *T* and *S* phases precipitate in the substructure of the studied alloy. The appearance of these equilibrium precipitates is the main reason for quenching sensitivity.

2) For water-quenched alloys, the main aging precipitates of T6 sample are GP zones and η' phases, and of T7 sample are η' and η phases, while those of RRA sample are GP zones, η' and η phases. Precipitation differences lead to the differences in their hardness behaviors.

3) For air-quenched alloys, the precipitates are coarser than those of water-quenched ones, plus that fewer solute atoms are obtained during quenching, thus lower aging hardening effect is achieved. Coarser precipitates in T7 and RRA treated alloys lead to further decrease in hardness and increase in quench sensitivity.

4) The DSC results are consistent with TEM observations. The dissolution and subsequent transformation behaviors of T7 and RRA aged samples are similar, but that of T6 have three particular characteristics: the T_P of peak A is the lowest, peaks A and B are simultaneous, and the enthalpy of region II is the highest.

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淬火析出对 Al-Zn-Mg-Cu 合金时效行为的影响

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摘 要:采用硬度测试、光学显微镜(OM)、透射电镜(TEM)和差示扫描量热法(DSC)研究淬火和时效(T6、T7、 RRA)对 Al-Zn-Mg-Cu 合金微观组织的影响。研究发现水淬合金经 T6 时效后的硬度最高。T7 和 RRA 时效后样 品的淬火敏感性相当,较 T6 时效的高 1.2%。TEM 观察表明,合金的淬火敏感性主要是由缓慢冷却时非均匀析出 引起的。大量 η 相在再结晶晶粒内的 Al₃Zr 弥散粒子和(亚)晶界上形核,而 S 和 T 相在有高密度位错和缺陷的亚 结构区生成。时效后,平衡 η 相周围的 η'相更加粗大。经 T6、T7、RRA 处理后,这些析出相的尺寸和形貌呈现 出不同的特征。DSC 结果与 TEM 观察结果一致。T6 态的 DSC 曲线和 T7、RRA 态的不同,反映了不同的微观组 织。

关键词: Al-Zn-Mg-Cu 合金; 淬火; 淬火敏感性; 时效; 析出

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