Precipitates in 2099 Alloy

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Mechanical properties and microstructure of 2099 alloy in T6 and T8 temper were investigated by tensile test and TEM observations; moreover, the role of the Mg and Zn elements in 2099 alloy was studied. The results showed that the dominant precipitates in T6 peak aged stage of 2099 alloy were δ' , θ' and T₁ phase; while in T8 peak aged stage the precipitates contained δ' , T₁ phase and a small volume fraction of θ' phase. For 2099 alloy, the pre-stretching prior to aging promoted the precipitation of T₁ phase, thus improved aging strengthening. In T6 condition Mg addition alone promoted the precipitation of GP zone and θ' phase while Zn facilitated the formation of T₁ phase; the addition of Mg together with Zn stimulated the precipitation of T₁ phase obviously, and suppressed the coarsening progress of δ' phase.

Keywords: heat treatment; tensile properties; microstructure; pre-stretching; 2099 alloy

1. Introduction

Compared with traditional 2XXX and 7XXX series aluminum alloys, the Al-Li alloys offer lower density, higher strength, and superior stiffness to weight ratio as well as good weldability. It may provide up to 10~25pct weight savings when using on certain aircraft structures, and can improve both the weight efficiency and service performance as well, while the manufacturing and maintenance become much easier and its cost was far lower, compared with composite materials. All these advantages make Al-Li alloys the most competitive high-strength structural materials in aircraft industry during 21st century^[1]. 2099 alloy belongs to the third generation high performance aluminum lithium alloy developed by Alcoa Corporation; research indicates that it possesses low planar anisotropy, superior corrosion resistance and excellent damage tolerance^[2]. Due to this, 2099 alloy has been applied to the fuselage structural components of A380^[3]. 2099 alloy is a class of Al-Cu-Li series alloy containing minor Mg and Zn. Related articles is mainly concentrated on mechanical properties, fatigue crack propagation and fracture toughness. The mechanisms and effects of Mg and Zn on its microstructure and properties have not been interpreted detailedly in public reports. The objective of this study mainly is to investigate the tensile properties and microstructure of 2099 alloy in T6 and T8 conditions, the role of Mg, Zn micro-alloying in 2099 alloy is also investigated in detail.

2. Experimental Procedure

The experimental alloys with the compositions showing in Table 1 were cast as $\Phi 100 \times 200$ mm billets in argon atmosphere. The billets were homogenized at 530°C for 32h, scalped and extruded into $\Phi 12$ mm rods. The rods were solution treated at 540°C for 40min, followed by cold water quench quickly. The tensile specimens were cut from longitudinal direction of the rods. Some

specimens were subsequently aged at 175° C (T6), others stretched by 3% and then aged at 150° C (T8).

Tensile tests were performed on css-44100 Electronic Universal Testing Machine. A strain rate of 2×10^{-3} m/min was applied. Thin foils for TEM observations were twin-jet electronpolished in 3:1 (volume ratio) methanol/nitric acid solution cooled to-25 °C. Observations were made under various imaging conditions using a TecnaiG² 200 microscope operated at 200 kV.

rable i Analyzed composition of the experimental anoys (mass/o)								
Alloy No.	Cu	Mn	Mg	Zn	Li	Be	Zr	Al
1# (2099)	2.60	0.30	0.25	0.75	1.75	0.0001	0.09	Bal
2# (Zn-free)	2.53	0.30	0.21	-	1.72	0.0001	0.09	Bal
3# (Mg-free)	2.61	0.30	-	0.77	1.77	0.0001	0.09	Bal

Table 1 Analyzed composition of the experimental alloys (mass%)

3. Results

3.1 Tensile properties

Figure 1 shows the tensile properties/aging time curves for the three experimental alloys at T6 and T8 conditions. As shown in Fig.1(a), 2099 alloy (alloy 1) exhibits much more rapid and pronounced age-strengthening behavior than alloys 2 and 3 in T6 condition, with the highest tensile strength of 560 MPa after aging for 40h. Alloy 2(Zn free) and 3(Mg free) reached peak strength aged for 56h and 64h, with the tensile strength of 538 MPa and 505 MPa, respectively. It can be seen that the minor content of Mg and Zn substantially enhanced the tensile strength of 2099 alloy, accelerated the age response and improved the ageing strengthening effect.

The tensile properties of the three experimental alloys at T8 condition are presented in Fig.1 (b). The changes of tensile properties with aging time for the three alloys at T8 condition show similar tendency as T6 condition. Alloy 1 achieved the peak strength aged for 30h, with the tensile strength of 625 MPa, about 65 MPa higher than T6 condition. Alloy 2(Zn free) and 3(Mg free) gained peak strength aged for 40h and 50h, with the tensile strength of 616 MPa and 598 MPa, which is 74 MPa and 94 MPa higher than T6 condition, respectively. It is evident that the pre-stretching prior to aging can improves the peak strength and accelerates the ageing response.



Fig. 1 Tensile properties vs ageing time curves for the experimental alloys in T6 and T8 conditions (a) T6 (175°C) (b)T8 (3% pre-stretching +150°C)

Compared the peak strength of the three alloys between T6 and T8 conditions, the difference in their strength at T6 condition is more remarkable than that at T8 condition. At T6 peak-aged condition the differences in strength between alloy 1 and 2 and between alloy1 and 3 are 22 MPa and 55 MPa, respectively, while at T8 condition, these differences decreased to about 9 MPa and 27 MPa correspondingly, suggesting that even though the pre-stretching before aging enhanced the strength of the alloys, it reduced the difference in strength of the three alloys and weakened the alloying effect of minor Mg and Zn on the age-hardening process.

3.2 Microstructure

3.2.1 Microstructure at T6 condition



Fig. 2 TEM images of 1#, 2#, 3# alloys under-aged in T6 condition (a) 1# alloy, b=<001> α ; (b), (c) 2#alloy, b=<001> α ; (d) 3#alloy, b=<001> α



Fig. 3 TEM images of 1#, 2#, 3# alloys peak-aged in T6 condition
(a), (b), (c) 1# alloy; (d), (e), (f) 2# alloy; (g), (h) 3# alloy
(a), (b), (d), (e), (g):b=<01>α (c), (f), (h): b=<112>α

The TEM images of the three alloys at under-aged stage in T6 condition are showed in Figure 2. The images indicate that the fine dispersion δ' phase is the major precipitates at the early aging stage, and it can be seen that the δ' phase in alloy 1 is very small compared with the others. The steaks in corresponding SAED patterns of alloy 1 and BF image of alloy 2(Fig.2(c)) may be induced by G.P Zone, which have not been observed in alloy 3.

Figure 3 presents the TEM images of the three alloys at peak-aged stage in T6 condition. It can be seen from Figure 3 that T1 plates are the prominent precipitate in alloy 1 with a large quantity of fine δ' phase and a small number of θ' phase. The T1 phase in alloy 2 (Zn free) is obviously decreased compared with alloy 1, a small quantity of θ phase was also observed. Thus it can be concluded that minor addition of Zn together with Mg is favored to the precipitation of T1 phase. A large quantity of T1 can also be seen in alloy 3(Mg free), but θ' phase was not observed, indicating that the addition of Mg had positive effect on θ' phase precipitation^[4]. The precipitates observed in under-aged and peak-aged stages of the experimental alloys at T6 condition are concluded in Table 2.

Alloy No	Under-aged	Peak-aged		
1# (2099)	fine δ' phase, GP zone	δ' , T1 phase, a small quantity of θ'		
2# (Zn-free)	δ' phase, GP zone	$\delta',$ a small quantity of θ' and T1 phase		
3# (Mg-free)	δ' phase	δ' phase, more T1 phase		

Table 2 The precipitates of experimental alloys under-aged and peak-aged inT6 condition

3.2.2 Microstructure at T8 condition



Fig. 4 TEM images of 1#, 2#, 3# alloys peak-aged in T8 condition
(a), (b) 1# alloy; (c), (d) 2# alloy; (e), (f) 3# alloy
(a), (c), (e): b=<001>α; (b), (d), (f):b=<112> α

The microstructure and precipitation of the three alloys at T8 condition are presented in Figure 4. The δ' phases and T1 phases of the three alloys in T8 condition are much smaller and more dispersal compared with those in T6 condition. It can be concluded that the pre-stretching prior to ageing stimulated the precipitation of T1 phase and slowed down the coarsening process of secondary phases. A large number of δ' phases and fine dispersal T1 phases as well as a few θ phases were observed in alloy 1. Heterogeneous nucleation of δ' phases enveloped on θ' phases and

formed δ'/θ' composites precipitations (Fig.4 (a)). Spherical δ' phase and fine T1 plates can also be seen in alloy 2 and alloy 3, while the density of Tl plates of alloy 2 is lower than that of alloy 1.

4. Discussion

The precipitation sequence and strengthening phases of Al-Cu-Li series alloys are strongly depended on Cu/Li ratios and micro-alloying elements. At high Cu/Mg ratios alloy it is easy that numerous Cu-Mg-vacancy co-clusters and Cu-Mg atom-pairs formed after solution treatment as a result of the strong interaction between Mg, Cu atoms and vacancies^[5]. At the early aging stage, the co-clusters and atom-pairs lead to strong segregations of solute atoms, which is favoured to the formation for G.P Zone^[6]. As seen from Fig.2, alloy 1 and alloy 2 containing Mg precipitated fine uniform G.P Zone at the early stage, and then gradually transformed into θ'/θ' phases at peak aged stage (Fig.3). Hence, it may come to this conclusion that the trace amount addition of Mg will stimulate the precipitation of G.P Zone and θ' phase. In alloy 3 (free Mg) no θ' phases were observed. This means that Mg additions may be essential for the formation of G. P. $Zone/\theta'$ phases. Figure 3 also shows that there are more T1 phases in alloy 1 and alloy 3 containing Zn than in alloy 2 (Zn free), indicating that the addition of Zn, especially, combined addition of Zn and Mg had a positive effect on the formation of T1 phase^[7]. However, the mechanism of Zn additions for the T1 precipatation in the Li containing alloy is not clear. The role of Zn additions may be similar to that of Ag. It has been suggested that a combined addition of Ag and Mg to Al-Cu-Li alloys promoted the uniform dispersion of the T1 phase. The 3DAP analysis results have clearly conformed that T1 phase is associated to Ag and Mg atoms which were segregated at the Tl/ α interfaces. It is possible that apart from decreasing the stacking fault energy of Al-matrix as the result of Zn atoms segregated at <111> plane, Zn atoms formed a great deal of Zn-Mg atom-pairs and Zn-Mg-vacancy co-clusters at the early stage, which provided the priority nucleation sites for T1 phase^[8~10].

While in T8 condition, the pre-stretching increased the number of matrix dislocations, which provided massive heterogeneous nucleation sites, thereby facilitating the formation of T1 phase $^{[11\sim12]}$. Moreover, it also accelerated the nucleation kinetics and restrained the growth of T1 phase. T1 phase nucleated and grew at the cost of δ' phase and θ' phase $^{[13]}$. Therefore, it exhibited a competitive precipitation relationship between θ' phase and T1 phase. The dislocations induced by pre-stretching leaded to the annihilations of vacancy. It was harder for Mg and Zn to form the co-clusters with vacancy, and hence weaken the influence of Mg and Zn additions to the precipitation of the alloys in a certain extent.

5. Conclusion

1) The major precipitates of 2099 alloy in T6 and T8 condition are δ' and T1 phase with a small quantity of θ' phase

2) In T6 condition, the Mg additions, in experimental alloy promoted the formation of G.P Zones at early stage, and then gradually transformed into θ''/θ' phase at peak aged stage. The Zn additions facilitated the precipitating of T1 phase and restrained its coarsen. The combination role of Mg and Zn stimulated the precipitation of T1 and θ' phase and lead to fine and uniform dispersal precipitations.

3) In T8 condition, the pre-stretching prior to aging significantly promoted the precipitation of T1 phase due to the dislocation being as heterogeneous nucleation sites preferentially, and reduced

the influence of Mg and Zn on the precipitation, and thus decreased the difference of the strengt between the three alloy at peak aged stage.

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