Improved Properties in Relation to Fine Precipitate Microstructures Tailored by Combinatorial Processes in an Al–Cu–Mg–Si Alloy

Fengjiao Niu¹ · Jianghua Chen¹ · Cuilan Wu¹ · Jing Wu¹ · Xiandong Xu¹ · Pan Xie¹ · Xiongwei Yu¹

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Abstract

The 2xxx series Al alloys have been widely used in aerospace industry owing to their high strength, good plasticity and superior formability. To ensure a good control of shape, the quenched alloy sheets require a small pre-deformation before artificial aging. However, this pre-deformation considerably deteriorates the mechanical strength of the Al–3.0Cu–1.8Mg–0.5Si (wt%) alloys due to the formation of unfavorable large-sized precipitates at dislocations. To tackle this issue, we designed a pre-aging process prior to the pre-deformation. The thermal–mechanical treatment, involving pre-aging, pre-deformation and subsequent aging, markedly enhanced the ultimate tensile strength up to 521 MPa compared to that (448 MPa) of the alloy without pre-aging. Microstructure characterization revealed that the fine precipitates (~ 2 nm) with a uniform dispersion were promoted within the Al matrix, which in turn partly suppressed the formation of the unfavorable large-sized precipitate (~ 100 nm). Our findings provide a new clue for designing stronger Al alloys with age-hardenability.

Keywords Aluminum alloys · Thermo–mechanical treatment · Precipitation · Age-hardenability

1 Introduction

Al–Cu–Mg(–Si) alloy sheets have been widely used in aerospace industry owing to their high strength, good plasticity and superior heat resistance [1]. The production for the Al–Cu–Mg(–Si) sheets requires a straightening process to control the shape of as-quenched sheets, which are T8 process involving pre-deformation and subsequent artificial aging. Compared with the traditional aging (T6 process), T8 process usually results in the change of the microstructure and mechanical properties of materials. This is because deformation-induced dislocations can not only serve as a fast diffusion channel for solute atoms, but also trap solute atoms, which lead to a markedly different precipitation process during the subsequent aging process [2–4]. This difference was found to be either favorable or unfavorable to the mechanical performance of aluminum alloys due to the

stronger or weaker strengthening effect of the dislocation-induced precipitates, respectively [5, 6]. On the one hand, more nucleation sites at dislocations result in a greater density of stronger precipitates and finally increase the peak hardness in Al–Cu–Mg alloys [6–8]. On the other hand, for Al–Cu–Mg–Ag alloys, high density of deformation-induced dislocations increases the density of the θ' phase but decreases the density of the effective strengthening Ω phase, which lead to loss in peak [2, 5, 9].

For Al–Cu–Li alloys, the pre-deformation affects the macroscopic performance by affecting the competitive relationship of T1 and θ' phase, the result of which is strongly related to the alloy composition [10]. For Al–Zn–Mg–Cu alloys, the tensile strength of the aged 7050 alloy with 2.3% pre-stretching is lower than that of samples without pre-stretching, resulting from the precipitation of rod-shaped η phase induced by the dislocation [11]. But the ultrafine grained (UFG) 7075 alloy made by equal-channel angular pressing (ECAP) or cryo-rolling process can obtain higher tensile strength without large loss of ductility because of the higher densities of shearable Guinier–Preston (GP) zones and dislocations in the UFG samples [12, 13]. For Al–Mg–Si alloys, the introduction of pre-deformation not only changes the density of precipitates, but also changes the types of precipitates

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✉ Cuilan Wu
cuilanwu@hnu.edu.cn

¹ Center for High-Resolution Electron Microscopy, College of Materials Science and Engineering, Hunan University, Changsha 410082, China

in the undeformed regions due to loss of solute atoms [14]. Furthermore, semiquantitative calculations showed that precipitation strengthening is weakened due to the structural changes in precipitates even when the total yield strength of the 10% pre-deformed Al–Mg–Si alloys is slightly improved compared to the alloy without pre-deformation [15]. Therefore, there is no certain statement for the effect of pre-deformation on the performance and precipitation for aluminum alloys with varied compositions.

For Al–3.0Cu–1.8Mg–0.5Si (wt%) alloys, the main precipitates are the Si-modified Guinier–Preston–Bagaryatsky (GPB) zones generated during the conventional T6 aging process [16]. Our previous study illustrated that the introduction of 6% pre-deformation results in the dislocation-induced precipitation of the zig–zag composite precipitate (CP) with a large size and decreases the number of Si-modified GPB zones [12]. So cold-rolling deformation of 6% prior to artificial aging leads to a degradation in the mechanical properties of an Al–Cu–Mg–Si alloy with a low Cu/Mg ratio compared to the alloys without pre-deformation [17]. To improve the mechanical properties, microstructure must be modified by reducing the amount of the zig–zag composite precipitates or tuning the zig–zag composite precipitates at a finer size.

Gratifyingly, it has been proved that the mechanical properties of aluminum alloys can be improved by applying a pre-aging treatment prior to deformation and subsequent aging, which was termed thermo-mechanical treatment (TMT) [18–21]. The redistribution and combination of the solute atoms during the pre-aging treatment improved the tensile properties by inhibiting the dynamic recovery and consequently raising the dislocation density during the large deformation (with strain > 75%) [19, 22], or modifying the precipitation behavior during the final artificial aging [23]. The pre-aging prior to the 1% stretching ensured a strong Mg–Ag co-clustering and hence promoted a dense precipitation of the Ω phase in the deformed Al–Cu–Mg–Ag alloy, which leads to a higher strength than that of alloy without pre-aging treatment [24]. Nevertheless, little is known on the influence of pre-aging treatment on the mechanical properties of the Al–Cu–Mg–Si alloys with a small deformation (with strain < 10%).

In the present work, pre-aging treatments were applied to modify the precipitation sequence during the final aging process for Al–Cu–Mg–Si alloys processed by 6% deformation. Through controlling the aggregation and distribution of alloying elements before cold rolling, we successfully obtained Al–Cu–Mg–Si sheets with a remarkable enhancement in strength without obvious decrease in ductility. The influence of the different pre-aging treatments, including natural aging and artificial aging, on the precipitation behavior during the subsequent deformation and aging process is investigated.

2 Experimental

Ingots with a chemical composition of Al–3.0Cu–1.8Mg–0.5Si–0.15Zn–0.1Ti–0.1Fe (wt%) were prepared by arc melting under an argon atmosphere. The resultant ingots were homogenized at 510 °C for 24 h in an air-cycling furnace, then hot-rolled at 450 °C from 20 to 5 mm and further cold-rolled to sheets of 2 mm in thickness after multiple passes.

Table 1 displays the detailed processes of different heat treatments in the present work. The cold-rolled samples were firstly solution-treated (ST) at 535 °C for 1 h, water-quenched (WQ) and then treated by different aging processes. The WQ samples are divided into four groups according to Table 1. Samples of the first group were artificially aged (AA) immediately at 180 °C in a constant temperature oil bath furnace (hereafter called T6 heat treatment). Samples of the second group underwent immediately 6% cold-rolling (CR) deformation, followed by the same aging treatment at 180 °C, which is named T8 temper. Samples of the last two groups firstly underwent pre-aging treatment, including natural aging (NA) for 7 days or artificial aging (AA) at 200 °C for 1 h, and then underwent 6% deformation followed by the same aging at 180 °C, which can be called NA7d-TMT and AA1h-TMT process, respectively. Then, the hardness measurements were carried out for different samples. The Vickers hardness was measured with a load of 4.9 N and a dwell time of 10 s. Each hardness value was the average value of at least 5 indentations. The tensile properties of peak-aged samples were also characterized. The tensile samples were machined as per the ASTM Standard E517-00 with longitudinal axis parallel to the cold-rolling direction. Each data of the tensile properties is the average value of at least 4 samples with the same aging history.

Samples for transmission electron microscopy (TEM) observations were prepared using a twin-jet electro-polishing device. TEM observations were carried out using a FEI Tecnai F20 TEM with an acceleration voltage of 200 kV and equipped with a high-angle annular dark-field (HAADF) detector. The beam convergence semi-angle and collection angle range for scanning TEM (STEM)

Table 1 Detailed process of the heat treatments used in this article

Heat treatment	Detailed process
T6	ST + WQ + AA at 180 °C
T8	ST + WQ + 6% CR + AA at 180 °C
NA7d-TMT	ST + WQ + NA for 7 days + 6% CR + AA at 180 °C
AA1h-TMT	ST + WQ + AA at 200 °C for 1 h + 6% CR + AA at 180 °C

observations were 10 mrad and 36–200 mrad, respectively. To improve the image quality, the recorded images were processed by Fourier-filtering to remove high-frequency noises.

3 Results and Discussion

3.1 Mechanical Properties

Figure 1a shows the single-stage age-hardening curves of the samples pre-aged at room temperature (RT) and 200 °C, respectively. The hardness value of the naturally aged samples increased with NA time and peaked at aging for 7 days. By contrast, for the latter case with 200 °C-aging, the hardness value increased rapidly at the early aging stage and reached a peak plateau after aging for 1 h.

The age-hardening responses of the samples treated by TMT, T6 temper and T8 temper are shown in Fig. 2b. The alloy reaches a peak-hardness value of 153 HV for 48 h in the T6 aging process. The time to reach the peak hardness is shortened to 18 h for the T8 aging process, but the peak hardness then decreases to approximately 140 HV. For the NA7d-TMT process, there is a short-term hardness drop at the early stage of subsequent aging, and then, the hardness gradually increases to the peak-hardness value of 163 HV at 24 h. For the AA1h-TMT process, the hardness reaches its peak of 157 HV quickly at 9 h.

The hardness value and tensile properties, including yield strength ($\sigma_{0.2}$), tensile strength (σ_b) and elongation (ϵ), of the peak-aged samples are summarized in Table 2. The hardness obviously decreases for the T8-treated samples but increases for the TMT-treated samples, as compared with the T6-treated samples. The yield strength and ultimate tensile

strength of the NA7d-TMT samples increase by 56 MPa and 73 MPa, respectively, compared to those of T8 samples without pre-aging treatment.

3.2 Microstructure Characterization

3.2.1 T6 and T8 Tempers

Figure 2 displays the HAADF-STEM images of the peak-aged samples treated by T6 and T8 tempers, respectively. In T6 peak-aged samples, the uniformly dispersed Si-modified GPB zone with needle-like morphology is the main strengthening precipitates although the rod-like S phase can also be observed frequently. For T8 peak-aged samples, the zig-zag composite precipitates induced by dislocations become the main precipitates except for a small amount of Si-modified GPB zone. The zig-zag composite precipitates are confirmed to have a long-range disorder, whereas short-range ordered S phase, C phase and GPB zone are observed [17]. As the main constitute of the zig-zag composite precipitates, S phase always has a thickness of over 1.5 nm. It has been revealed that the properties of alloy are directly related to the morphology and distribution of the precipitates [25]. Moreover, for a given volume fraction and number density of precipitate plates, an increase in precipitate plate aspect ratio can lead to a substantial increase in any increment in critical resolved shear stress required to promote dislocation motion within the precipitate distribution [26, 27]. It can be concluded that larger-sized and a smaller amount of precipitates are detrimental to the properties for aluminum alloys with age-hardenability. Therefore, the zig-zag composite precipitates of T8 samples are prone to coarsen and line up to longer than 100 nm, which results in the deterioration of hardness and tensile strength [17].

Fig. 1 **a** Hardness–time curves of the Al–3.0Cu–1.8Mg–0.5Si alloy pre-aged at RT and 200 °C, and samples marked with arrows are selected to undergo the TMT processes, **b** Vickers hardness plotted against aging time curves of the samples treated by T6, T8 temper and TMT processes aging at 180 °C

Fig. 2 HAADF-STEM images of the peak-aged samples treated by T6 temper **a, b**, T8 temper **c, d**

Table 2 Mechanical properties of the peak-aged samples with different heat treatments

Heat treatment	Peak-aging time (h)	Peak hardness (HV)	$\sigma_{0.2}$ (MPa)	σ_b (MPa)	ϵ (%)
T6	48	153	411	469	9.9
T8	18	140	429	448	7.8
AA1h-TMT	9	157	454	500	7.3
NA7d-TMT	24	163	485	521	8.3

3.2.2 TMT Processes

As mentioned above, the samples aged at RT for 7 days and samples aged at 200 °C for 1 h were used to undergo

the NA7d-TMT and AA1h-TMT processes, respectively. Because it is impossible to reveal the NA clusters using the TEM characterization, the microstructure of NA7d samples is not shown here. Figure 3 displays the microstructure of the samples pre-aged at 200 °C for 1 h, in which the main precipitates are almost the precursors of needle-like Si-modified zones. It is worth noting that the precipitation sequence of the alloys pre-aged at 200 °C is as same as that of the alloys aged at 180 °C [17].

Figure 4 demonstrates the microstructure of the peak-aged samples treated by AA1h-TMT process and NA7d-TMT process. For AA1h-TMT samples, the zig-zag composite precipitates and uniformly distributed Si-modified GPB zones were observed everywhere, but the length of the zig-zag composite precipitates is always smaller than 50

Fig. 3 Bright-field TEM image **a** and HAADF-STEM image **b** of the samples pre-aged at 200 °C for 1 h. The inset in **b** showing the structure of a precursor

Fig. 4 HAADF-STEM images of the peak-aged samples treated by AA1h-TMT process **a–c** and NA7d-TMT process **d–f** viewed along the $[001]_{Al}$ direction

nm and the thickness of S phases is always smaller than 1.5 nm. Furthermore, compared with the T8 samples, there are more Si-modified GPB zones in the AA1h-TMT samples, even in the vicinity of the zig-zag composite precipitates as shown in Fig. 4b.

The peak-aged samples treated by NA7d-TMT process contain many zig-zag composite precipitates, rod-like S phase and uniformly distributed Si-modified GPB zone. The rod-like S phases, which are lined up as shown in Fig. 4d–f, must be developed from the zig-zag composite precipitates formed in the earlier aging stage, since the rod-like S phase is supposed to be more stable [28, 29]. As such, most of the zig-zag composite precipitates in the NA7d-TMT peak-aged samples are broken and their sizes become smaller, which is beneficial for the mechanical properties of the alloys. So the mechanical properties of the NA7d-TMT peak-aged samples are better than those of the T8 samples, as shown in Fig. 1 and Table 2.

To reveal the size and distribution of precipitates induced by dislocation in the peak-aged samples with different processes, the low-magnification HAADF-STEM images are

provided in Fig. 5. The zig-zag composite precipitates of the TMT samples are more evenly distributed and are smaller in size than that of T8 samples. The zig-zag composite precipitates which are longer than 200 nm can be observed in T8 samples frequently, while the length of the zig-zag composite precipitates in TMT samples is always less than 50 nm. The distribution of the dislocation-induced precipitates in NA7d-TMT peak-aged samples is most uniform among all samples with different treatments.

To compare the precipitation behaviors clearly, the main strengthening precipitates of the peak-aged samples treated by different aging treatments are listed in Table 3. The Si-modified GPB zone plays an important role in the strengthening for the T6-treated samples. The T8-treated samples are mainly strengthened by the zig-zag composite precipitates (CPs) with a large thickness and length and a small amount of Si-modified GPB zones. For the peak-aged samples of AA1h-TMT process, there are some zig-zag CPs with a smaller length and a large amount of the Si-modified GPB zones induced by the precursors. For the peak-aged samples of NA7d-TMT process, there are three types of precipitates,

Fig. 5 Low-magnification HAADF-STEM images of the peak-aged samples treated by T8 temper **a, b**, AA1h-TMT process, **c, d**, NA7d-TMT process, **e, f** viewed along the $[001]_{\text{Al}}$ direction

Table 3 Precipitates of the peak-aged samples with different heat treatments

Heat treatment	Main precipitates	
	Dislocation-induced precipitates	Dislocation-free precipitates
T6	–	Si-modified GPB zones
T8	The coarse CPs (~ 100 nm)	Si-modified GPB zones
AA1h-TMT	A small amount of CPs (~ 50 nm)	A large amount of Si-modified GPB zones
NA7d-TMT	A small amount of CPs (~ 50 nm), some rod-like S phase	A large amount of Si-modified GPB zones

including zig–zag CPs with a smaller length, rod-like S phase and Si-modified GPB zones. The density of precipitates with small sizes in the NA7d-TMT peak-aged samples is the largest among the three groups of samples with pre-deformation, so the NA7d-TMT peak-aged samples obtain the highest peak-hardness and better tensile properties.

3.3 Discussion

In the present work, the differences in macroscopic properties of the same alloys with different processes may come from dislocation strengthening, grain boundary strengthening and precipitation strengthening. The amount of deformation is so small that the change in grain size is not obvious [30]. Therefore, the grain boundary strengthening will not be considered in this article.

As mentioned above, pre-aging prior to deformation resulted in different precipitation behaviors and age-hardening curves during the final aging process. It is necessary to analyze the diffusion path of the solute atoms to explore the precipitation mechanisms. The vacancy binding energies of alloying elements have an impact on the thermodynamics and kinetics of the precipitation during the aging process. In Al–Cu–Mg–Si alloys, the vacancy binding energies for Si, Mg and Cu are 0.08, –0.02 and 0.04 eV, respectively [31], which means that the Mg-rich clusters would be easy to form rapidly during the aging process for the as-quenched samples. Si and Cu atoms gradually segregate to the Mg-rich clusters, leading to the formation of NA clusters during the natural aging process [32, 33] and the precipitation of the precursor of Si-modified GPB zones in the early stage of the artificial aging process [16]. For the T6 process, the Si-modified GPB zone and its precursor can form in the early stage of aging and few of S phases appears in the peak stage of aging. The precipitation of Si-modified GPB zone precedes that of S phase, which is consistent with previous studies [16, 34]. A large number of uniformly dispersed needle-like Si-modified GPB zones play an important role on the high strength and excellent plasticity of T6-treated samples.

For the deformed samples without the pre-aging treatment, the free paths of dislocations produced by deformation are very long. After the small deformation (6% cold rolling), dislocations are formed in the matrix. It is worth

noting that the inevitable natural aging before artificial aging can be suppressed by the dislocations induced by deformation [35]. The solute atoms and vacancies are easily segregated near the dislocations to promote the precipitation of zig–zag composite precipitates during the final aging process. A large number of Cu, Mg, Si atoms and vacancies are trapped by the dislocations, which results in the decrease in solute atoms for the nucleation of Si-modified GPB zones, so the number of uniformly dispersed of Si-modified GPB zones is largely reduced compared to the T6-treated alloys. On the other hand, the zig–zag composite precipitates are much coarser and longer than the needle-like Si-modified GPB zones, so the strengthening effect is not as good as that of Si-modified GPB zone, leading to the lower peak-hardness value, tensile strength and elongation of T8-treated samples than those of T6-treated samples.

For the NA7d-TMT samples, it can be speculated that the natural aging clusters or the precursors of Si-modified GPB zone must affect the movement and reaction of dislocations during the subsequent cold rolling. Owing to the pinning effect of uniform small precipitates or clusters [36] and their inhibitory effect on dynamic recovery during deformation [37–39], a higher density of dislocations should be uniformly stored in the as-rolled TMT alloy, which further provides larger amount of nucleation sites and reduces the size of dislocation-induced precipitates. Besides, it was believed that the unstable NA clusters will dissolve into the matrix, and the stable NA clusters promote the precipitation of the Si-modified GPB zone by acting as nucleation sites of the strengthening precipitates during the subsequent aging process [40, 41]. The decrease in hardness at the initial stage of the final artificial aging (as shown in Fig. 1b) illustrates that some clusters in the NA7d-TMT samples are first dissolved and then promote the precipitation of the Si-modified GPB zones. So the peak-aging time of NA7d-TMT samples is longer than that of T8 process due to the dissolving process. The peak-aging time is so long that the zig–zag composite precipitates become more mature, leading to the formation of rod-like S phase. The more uniform distribution of dislocation-induced precipitates and a lot of Si-modified GPB zones greatly improve the strength of the alloy and slightly improve the elongation compared with the T8 samples.

For the AA1h-TMT samples, the precursors, forming in the process of pre-aging at 200 °C for 1 h, promote the precipitation of Si-modified GPB zone during the final aging process. So the hardness reaches its peak within a short period of time, and the number density of Si-modified GPB zone is supposed to be larger than that of T8 samples. At the same time, shorter aging time results in the smaller thickness and length of the zig-zag composite precipitates. Therefore, the number of Si-modified GPB zones in the AA1h-TMT samples is more than that of the T8 samples, while that of the large-sized zig-zag composite precipitates is more than that of NA7d-TMT samples. As such, the strength of the AA1h-TMT samples is higher than that of T8 samples, but lower than that of NA7d-TMT samples.

4 Conclusions

In the present work, through applying natural aging for 7 days or artificial aging for 1 h before 6% deformation, we modified the precipitation behaviors during the final aging process and improved the mechanical performance. The main conclusions can be drawn as follows:

- (1) Compared to the T6 temper, the peak-hardness value and tensile strength of the alloys are obviously reduced through the T8 temper and significantly improved through the TMT processes with pre-aging treatment.
- (2) This reduction in the mechanical properties for T8-treated samples is ascribed to the precipitation of large-sized precipitates induced by dislocation and the decrease in fine needle-like Si-modified GPB zones.
- (3) This reduction in the mechanical properties for T8-treated samples is ascribed to the precipitation of large-sized precipitates induced by dislocation and the decrease in fine needle-like Si-modified GPB zones.

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