Revisiting the Hierarchical Microstructures of an Al–Zn–Mg Alloy Fabricated by Pre-deformation and Aging

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Received: 7 January 2020 / Revised: 22 March 2020 / Published online: 17 June 2020
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Abstract

Pre-deformation before aging has been demonstrated to have a positive effect on the mechanical strength of the 7N01 alloy in our previous study, which is rather different from the general negative effects of pre-deformation on high-strength 7XXX aluminum alloys. In order to explain the strengthening mechanism relating to the positive effect, in the present study, the microstructure of the aged 7N01 alloy with different degrees of pre-deformation was investigated in detail using advanced electron microscopy techniques. Our results show that, without pre-deformation, the aged alloy is strengthened mainly by the η' type of hardening precipitates. In contrast, with pre-deformation, the aged alloy is strengthened by the hierarchical microstructure consisting of the GP- η' type of precipitates formed inside sub-grains, the η_p type of precipitates formed at small-angle boundaries, and the dislocation introduced by pre-deformation (residual work-hardening effect). By visualizing the distribution of the η_p precipitates through three-dimensional electron tomography, the 3D microstructures of dislocation cells are clearly revealed. Proper combinations of η_p precipitates, GP- η' precipitates and residual dislocations in the alloy are responsible for the positive effect of pre-deformation on its mechanical properties.

Keywords Aluminum alloys · Precipitates · Pre-deformation · Three-dimensional electron tomography (3DET) · Dislocation cell

1 Introduction

Al–Zn–Mg–(Cu) alloys are widely used in automobile and aerospace industries due to their high strength-to-weight ratios, good ductility and superior resistance to oxidation and corrosion [1, 2]. With proper thermal treatment, the Al–Zn–Mg–(Cu) alloys can achieve an ultrahigh strength (> 600 MPa) by means of the precipitation strengthening, which contributes 66–74% of the total strength of the alloy [3]. Many types of precipitates have been reported during thermal aging in Al–Zn–Mg–(Cu) alloys, such as the commonly reported Guinier–Preston (GP) zones [4], η' [5, 6] and η phases [7–9] and the recently reported GP- η_p , GP- η'

and η_p phases [10]. The main strengthening phases are different according to the alloy composition and heat treatment. For high-strength alloys with a high Zn(Cu)-to-Mg atomic ratio (> 2.4) such as the 7055 alloy, the dominant precipitates are the GP- η_p and η_p phases. However, in the middle-strength alloy with a relatively low Zn(Cu)-to-Mg atomic ratio (< 2.0) such as the 7N01 alloy, the dominant precipitates are the GP- η' and η' phases [11, 12].

Improving the performance of aluminum alloys to meet higher demands in automobile and aerospace application has always been a concern. An effective approach is to optimize the thermal process, such as double-stage aging or triple-stage aging treatment [13, 14]. However, a shortage of this approach is that it cannot modify the grain size and grain boundary type. Another effective method is severe plastic deformation (SPD), such as equal channel angular pressing (ECAP) [15], accumulative roll bonding (ARB) [16] and high pressure torsion (HPT) [17]. These methods form ultrafine sub-structure to enhance the mechanical performance of alloys via the interactions of dislocations and grain boundaries. Unfortunately, these techniques are unsuitable for practical application in

Available online at <https://link.springer.com/journal/40195>

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industry, as they always result in a rather low ductility due to the enormous deformation degree and the sample size is highly limited. In addition, the ultrafine substructures can easily be degraded during the subsequent high temperature aging. By combining the advantages of aging strengthening and plastic deformation, a more general and effective method of improving the mechanical properties of aluminum alloys is pre-deformation and aging treatment [18–20].

The effect of pre-deformation on artificial aging has been investigated in many heat-treatable aluminum alloys. A widely accepted view is that the presence of dislocations that are generated in the pre-deformation process greatly influence the subsequent precipitation process during aging treatment and therefore modifies the mechanical properties of alloys [21]. However, this effect can be positive or negative depending on the alloy composition and precipitate types. For example, in Al–Cu(Mg) alloys, pre-deformation could promote the precipitation of θ' (Al₂Cu) [22] or S (Al₂CuMg) [23–25] phase. Similarly, the precipitation kinetics of main strengthening precipitate, β'' (Mg₅Si₆), could also be accelerated in Al–Mg–Si alloys [26, 27].

In Al–Zn–Mg–(Cu) alloys (7XXX series), as reported in literatures, pre-deformation is more likely to have negative effects on the strength of this alloy, even though the deformation degree plays an important role as well. In AA7108 and AA7030 alloy, the samples with 10% pre-deformation exhibited lower yield strength than the T6 sample. The explanation for this was that the pre-deformation promotes the nucleation of the equilibrium phase η on the dislocation network, leaving less solutes in matrix for the formation of the main hardening phase η' [28]. Same results were also found in an Al–2.35 Mg–6.1 Zn alloy with 10% pre-deformation, but it was the η' precipitates which coarsened at dislocation cores [29, 30]. In 7050 alloy, it was found that the size of the η precipitates within grains increased with pre-deformation increasing from 0 to 5%, which led to the reduction in the mechanical strength [31]. Similar strength decrease by 10% was reported in 7050 alloy with 20% pre-deformation as well [20]. When larger pre-deformation (50%) was applied, the strength of the alloy maintained the same level as T6 treated sample in both 7050 [20] and 7A04 alloys [32]. Although the strength was significantly improved in a 80% pre-deformed 7075 alloy, due to the combination of increased rolling textures, high-density dislocations, nano-sized precipitates and refined grains, the plastic of the alloy is extremely low (~4%) [33] and cannot be used in industry.

Recently, significant improvement of yield and tensile strengths (≥ 55 MPa) with attainable elongation ($\geq 14\%$)

in the 7N01 alloy upon pre-deformation and aging was reported in our previous study [34]. However, the underlying microstructure in relation to the mechanical properties has not been revealed yet. Further, the microstructure evolution resulting from the increase of pre-deformation for this alloy is also unknown.

In this study, we aim to reveal the microstructures of the 7N01 alloy upon pre-deformation and aging using advanced transmission electron microscopy (TEM) techniques. Aged samples with different pre-deformation degrees, i.e. 20%, 40%, 60% and 80% height reductions by cold rolling, were carefully studied by TEM, high angle annular dark-field scanning TEM (HAADF STEM) and three-dimensional electron tomography (3DET). Firstly, the microstructure in traditional T6 sample was investigated as a reference. Then, the aged sample with 20% degree of pre-deformation was assessed carefully, since it had the optimum strength and elongation compared with T6 peak aging sample. Next, the influence of pre-deformation degree on the microstructure was investigated to explain the decrease of strength and elongation with increasing pre-deformation before aging in 7N01 alloy. At last, a theoretical model was proposed to clearly illustrate the microstructure evolution in deformed and aged 7N01 alloys.

2 Experimental

2.1 Material and Processing

A 10-mm-thick fully homogenized commercial 7N01 Al alloy plate with chemical composition listed in Table 1 is used in the present study. The plates were firstly solution-treated at 475 °C for 1 h and water-quenched (WQ) at room temperature. Then, the plates were cold-rolled from 10 to 8 mm, 6 mm, 4 mm and 2 mm, with 10% reduction per pass, corresponding to the pre-deformation degree of 20%, 40%, 60% and 80%, respectively. At last, the cold-rolled samples were subjected to aging at 120 °C for various time in an oil bath. For convenience, the aged samples are hereafter referred as Rx%-T6, where $x\%$ refers the pre-deformation degree. For example, the 20% pre-deformed sample with aging at 120 °C is termed as R20%-T6. For the purpose of comparison, traditional T6 treatments (aged at 120 °C in the present study) was conducted on the 10 mm thick commercial plate. In addition, Rx%-y h, where y is the aging time at 120 °C, indicates sample aged at 120 °C for y hours with pre-deformation $x\%$.

Table 1 Chemical composition of the Al–Zn–Mg alloy (wt%)

Zn	Mg	Fe	Si	Cr	Zr	Ti	Mn	Al
4.50	1.32	0.08	0.06	0.12	0.10	0.08	0.37	Bal.

2.2 Microstructure Characterization Techniques

Microstructure characterizations were conducted using a FEI Tecnai F20 TEM at Hunan University and FEI Titan ChemiSTEM TEM at Zhejiang University. 3DET experiment was carried out using the FEI Tecnai F20 operated in STEM mode. The tilt angle of the 3DET sample ranged from -60° to $+60^\circ$, with an interval of 1.5° . The reconstruction and rendering were performed using commercial software Inspect3D and Amira. To enable a more robust reconstruction, the more accurate but time-consuming simultaneous iterative reconstruction technique (SIRT), with iteration number of 30, was used.

The TEM specimens were prepared firstly by mechanical polishing to a thin foil under 100 μm and then by twin-jet electro-polishing in a methanol solution with 30% nitric acid below -25°C at an operating voltage of 20 V. Before TEM observation and 3DET image acquisition, the specimens were plasma cleaned by Fischione 1020 plasma cleaner to remove contaminations.

3 Results and Discussion

We reported previously that the middle-strength 7N01 alloy with a small pre-deformation before aging exhibited an optimum mechanical property after 100 h aging treatment at 120°C [34]. This report is pronouncedly different from those of published results that small pre-deformation led to a smaller peak hardness and strength in the high-strength Al–Zn–Mg–(Cu) alloys [20, 28–31]. In the following sections, a detailed microstructure characterization of the $R_x\%$ -100 h ($x = 20, 40, 60$ and 80) samples was carried out by TEM, HAADF STEM and 3DET to uncover the mechanism that influences the mechanical property.

3.1 Microstructure of T6 Sample

Figure 1 shows the microstructure of the T6 peak-aged ($120^\circ\text{C}/100\text{ h}$) sample. As can be seen from the low-magnification HAADF image (Fig. 1a), dense and fine precipitates are uniformly distributed in a grain, demonstrating a homogeneous microstructure. High-magnification HAADF image (Fig. 1b) shows the bright contrast of the precipitates, indicating Zn segregation in these precipitates. The atomic structure along $\langle 011 \rangle_{\text{Al}}$ is shown in the inserted image. According to literatures [11, 12], this phase is identified as the η' phase which is the main strengthening phase in the middle-strength 7N01 alloy. The statistics of diameters of the η' precipitates are shown in Fig. 1c, from which the average diameter is measured to be about 7.3 nm.

3.2 Microstructure of the R20%-100 h Sample

Figure 2a, b are the low- and high-magnification HAADF images of the R20%-100 h sample. Obvious dislocation cells (or sub-grains) resulting from pre-deformation are observed. In addition, there are two types of precipitates with different sizes in the R20%-T6 sample. The smaller precipitates are uniformly distributed in the sub-grains formed by dislocation walls, while the larger precipitates prefer to reside at the dislocation cell boundaries. To show this phenomenon more clearly, the TEM and HAADF images of the same area were recorded simultaneously, as shown in Fig. 2c, d. In principle, TEM is more sensitive to dislocation structure due to diffraction contrast, and HAADF image is more sensitive to element segregation or local sample thickness. In Fig. 2c, dislocation contrast can be seen, as indicated by the arrows. Meanwhile, small precipitates (black contrast) are uniformly distributed in the matrix. However, in Fig. 2d, not only small precipitates, but also large bright-contrast precipitates can be seen. Interestingly, it can be seen that the large precipitates are located in the vicinity of dislocations, which suggests the

Fig. 1 Microstructures of the T6 samples: **a** low-magnification HAADF STEM image of the T6 sample ($120^\circ\text{C}/100\text{ h}$); **b** HAADF STEM image of the precipitates in a grain. The inset is the atomic resolution HAADF STEM image of a precipitate. **c** Statistical chart of precipitates' diameter distribution. The electron beam is parallel to the $\langle 011 \rangle_{\text{Al}}$ zone axis

Fig. 2 Microstructures of the R20%-100 h sample: **a**, **b** low- and high-magnification HAADF images of the R20%-100 h sample showing the dislocation cells; **c**, **d** TEM and HAADF image of the same area showing the heterogeneous nucleation of precipitates on dislocations; **e** statistical chart of diameter of small precipitates and large precipitates

precipitates may be heterogeneously nucleated at the dislocations. During the plastic deformation, these precipitates can pin the dislocation and hinder its movement. The statistical chart of the diameter of small precipitates and large precipitates is shown in Fig. 2e. The average diameter of small precipitates inside the dislocation cells is measured to be about 5.7 nm, smaller than the 7.3 nm in T6 sample (Fig. 1c), which has not been reported in high-strength alloys with pre-deformation [20, 28–33]. The average diameter of the large precipitates is 14.3 nm, showing that dislocations cause the coarsening of precipitates.

Further atomic-resolution HAADF images of the two types of precipitates are shown in Fig. 3. Figure 3a shows the atom arrangement of one of the small-sized precipitates that are uniformly distributed in the matrix. No obvious periodic structure can be seen in the small precipitates. According to literatures [11, 12], this kind of precipitates may be identified as the GP- η' phase, rather than the η' phase in the T6 sample (Fig. 1b). The heterogeneously nucleated precipitates at dislocations were also characterized and shown in Fig. 3b. Utterly different periodic structure compared with the GP- η' phase in the matrix can be observed. The red box in Fig. 3b shows two unit cells of the precipitate with the perfect C_{14} -stacking order, and the measured c parameter of the unit cell along the $\langle 111 \rangle_{\text{Al}}$ direction was approximately equal to $4d_{\langle 111 \rangle_{\text{Al}}} (=0.935 \text{ nm})$. It can be identified as the η_p phase [11, 12], rather than the previously reported η phase or η' phase [28–31]. As η_p phase ($\text{Al}_2\text{Mg}_2\text{Zn}_8$) is Zn rich in composition, it was reported to prefer to nucleate in alloys with high Zn/Mg ratio, i.e. (Zn/Mg ratio > 2.4) [11, 12]. However, it is interesting to see that η_p phase formed in the low Zn/Mg atomic ratio 7N01 alloy (Zn/Mg = 1.3). Possible reason may be that the dislocations induced by pre-deformation provide a fast diffusion channel for the Zn atoms, which makes the Zn/Mg ratio in the dislocation region higher than 2.4 and promotes the precipitation kinetics of η_p phase. In this case, the Zn/Mg atomic ratio in the matrix may be less than 1.3, so the uniformly distributed GP- η' precipitates were formed.

Fig. 3 Atomic-resolution HAADF image of the R20%-100 h samples: **a** uniformly distributed precipitate in the matrix; **b** heterogeneously nucleated precipitate at dislocations. All images are recorded with the electron beam along the $\langle 112 \rangle_{\text{Al}}$ zone axis

It is believed from Fig. 2 that one of the factors affecting the mechanical properties of the R20%-100 h sample is the η_p “decorated” dislocation cells structure. However, the conventional 2D TEM image can hardly give the information about the 3D morphology of dislocation cells and the relationship with precipitates. Here, we try to characterize the dislocation cells by 3DET from specimen tilt series HAADF STEM images. In principle, dislocation contrast is very weak in HAADF STEM images. Therefore, most 3D reconstructions of dislocation are performed in TEM mode [35–38]. In the present study, the dislocations cells are surrounded by nanometer sized η_p phases that composed of heavier Zn atoms than Al matrix. Therefore, by displaying the 3D coordinates of the η_p precipitates in the specimen, the 3D morphology of the dislocation cells can be roughly described.

Figure 4 shows the rendered volume of nanoparticles. The size of the reconstructed volume is $2150\text{ nm} \times 2150\text{ nm} \times 249\text{ nm}$, and the overall morphology is

shown in Fig. 4a. The large-sized η_p precipitates and small-sized GP- η' precipitates were clearly displayed. Meanwhile, it can be seen in Fig. 4a that the large-sized η_p precipitates are not uniformly distributed throughout the sample. And some dislocation cell structures are formed as indicated by the blue arrows. Figure 4b is a projection observed along another angle as shown by the green–red arrows at the lower right corner of the image. The continuously arrangement of η_p precipitates, as indicated by the yellow arrow, forms a boundary of about 2000 nm in length, which is probably a grain boundary, since the average diameter of dislocation cells is about 300 nm. Figure 4c is a 3D sectioning of the reconstructed volume indicated by red arrow (Fig. 4a). Each image in Fig. 4c is a projection of 40-nm-thick slab at different depth along z-axis. The thickness and depth of each image are marked at the right side of the sub-figure. To clearly show the dislocation cell, the η_p precipitates near the boundary are marked by red curves. It can be seen that at the top slab, the area surrounded by the red curve is small, while

Fig. 4. 3DET images of the R20%-100 h sample: **a** overall morphology of the reconstructed volume; **b** projection of the tilted volume observed in another angles showing the grain boundary; **c**, **d** 3D sectioning of the arrowed area in **a** along z-axis; **c**, **d** displaying the red arrow marked area and the yellow arrow marked area, respectively

in the second top slab, the area increases. When it comes to the bottom slab, the area is the largest. This means that the sample thickness is at most half the diameter of this dislocation cell. Figure 4d is the 3D sectioning of the area indicated by yellow arrow in Fig. 4a. It is clear that the arrangement of the η_p precipitates is almost along a straight line, indicating the existence of a grain boundary. This is the first time that 3DET is used to visualize the dislocation cell structure by characterizing the heterogeneously nucleated precipitates on dislocations.

From Figs. 2, 3 and 4, it can be seen that hierarchical structures composed by dislocation cells, GP- η' precipitates and η_p precipitates are formed in R20%-100 h sample. Even though the strengthening effect of GP- η' precipitate is weaker than η' (T6) [4–9], the refined diameter of precipitates in R20%-100 h sample can make up the loss to some degree, so the precipitation strengthening contribution of the alloy should not decrease drastically comparing to that in the 7050 alloy with 20% pre-deformation [20]. Besides, the higher density of dislocations cells pinned by η_p precipitates also contribute to the alloy strength. Hence, it is reasonable that the strength increases in the middle-strength 7N01 alloy by 20% pre-deformation.

3.3 Influence of Pre-deformation Degree

As reported in our previous study, there was a slight decrease in ultimate tensile strength (UTS, from 460.5 to 443 MPa) and significant decrease in elongation (from 11.6 to 6.8%) when the pre-deformation degree increased from 20 to 80% for the Rx%-100 h ($x=20, 40, 60$ and 80) sample [34]. To explain the mechanical properties differences, the microstructure of Rx%-100 h samples ($x=40, 60$ and 80) was characterized, and the results are displayed in Fig. 5. With the increase in deformation degree, the dislocations in the matrix increases gradually. The average size of dislocation cells decreases and finally cannot be seen as pre-deformation increased to 80% (Fig. 5a, d, g). In addition, the small-sized precipitates and large-sized precipitates can also be seen in all samples, as indicated by green and red arrows in Fig. 5b, e, h. By employing atomic-resolution HAADF image characterization, the small- and large-sized precipitates are, respectively, recognized as the GP- η' and η_p phases, as with R20%-100 h sample. The average size of GP- η' phase in R80%-100 h is about 5.9 nm, similar to that in R20%-100 h sample. Therefore, it can be speculated that pre-deformation has little effect on the size of GP- η' phase. However, it significantly influences the diameters of the large-sized η_p precipitates, as shown in Fig. 5c, f, i. The average diameters of the η_p precipitates in the Rx%-100 h sample ($x=40, 60$ and 80) are, respectively, 16.2 nm, 17.7 nm and 19.8 nm, increasing with pre-deformation degree.

The coarsening of η_p precipitates consumes a large amount of solute atoms, resulting in a significant decrease in the volume fraction of small-sized precipitates (GP- η') in the sub-grains, which dramatically reduces the precipitation strengthening effect of the alloy as pre-deformation degree increases to 80%. However, larger pre-deformation will also increase the dislocation density in the matrix and modify the grain boundaries, which enhance the dislocation strengthening and grain boundary strengthening effect [33]. In this case, the strength of alloy is determined by the comprehensive effect of multiple strengthening mechanisms, including precipitation strengthening, dislocation strengthening, and grain boundary strengthening [39–41]. For the 7N01 alloy with large pre-deformation, this strength increase is insufficient to make up the decrease of precipitate strengthening. Therefore, the yield strength of the R80%-100 h sample is slightly lower than that of the R20%-100 h sample [34].

3.4 Microstructure Evolution of Pre-deformed Sample with Aging

When the pre-deformed alloy is subjected to thermal aging, the dislocation recovery and precipitation processes take place simultaneously in the matrix. For alloys with different pre-deformation, the dislocation density varies, which heavily influences local composition and may modify the precipitate types upon thermal aging.

To depict the microstructure evolution and mechanical property variation under different degrees of pre-deformation (20% cold rolling and 80% cold rolling), a diagrammatic sketch (Fig. 6) was drawn to help understand the process. After cold rolling (initial-stage aging), a large number of dislocations appeared and some form dislocation cells in the sample. For the large deformation (80%) treated sample, the dislocation cells are smaller than that in the small deformation (20%) treated sample due to the higher density of dislocations. Therefore, the R20%-T6 sample has higher capacity to dislocations formed during plastic deformation. This is why the elongation of the R20%-T6 sample is higher than that of the R80%-T6 sample.

At the early stage of aging process for the R20%-T6 sample, dislocations begin to recover and some η_p precipitates begin to nucleate on dislocations upon high temperature aging (Figs. 2, 3, 4, 5). With aging time extending, the η_p precipitates grow up gradually. At the later-stage of aging, η_p precipitates further grow up and the size of η_p precipitates increase, which makes the structure of dislocation cell more stable. For the R80%-T6 sample, there are high density dislocation cells in the initial stage of aging, and the nucleation sites of η_p precipitates are much more than those of the R20%-T6 sample. The inter-distance between neighboring η_p precipitates is shorter in the middle stage of aging, thus making it more like a uniformly distribution in the later-stage of

Fig. 5 HAADF images of the Rx%-100 h ($x=40, 60$ and 80) samples with different pre-deformation degrees: **a–c** R40%-100 h, **d–f** R60%-100 h, **g–i** R80%-100 h. **b, e, h** are enlarged images of corresponding boxed area marked in **a, d, g**. The statistical charts of precipitate diameter of the large precipitates are shown in **c, f, i**, respectively. The images were recorded with the electron beam along the $\langle 112 \rangle_{\text{Al}}$ zone axis

aging, as shown in Fig. 5g and h. In addition, larger strain also provides more energy for the growth of η_p precipitates, and therefore, the coarsening of η_p precipitate in the R80%-T6 sample is faster than that of the R20%-T6 samples.

4 Conclusions

In this study, the microstructure of the 7N01 alloy with different degrees of pre-deformation before aging treatment has been investigated. It is demonstrated that the dislocation

cell structure in the alloy resulting from deformation can be visualized by employing the 3DET method to image the 3D distribution of the dislocation-induced η_p precipitates in the Al matrix. From the results obtained, the following conclusions can be drawn.

1. In the pre-deformed samples, the homogeneously nucleated precipitates with smaller size are GP- η' precipitates, and those precipitates nucleated on the dislocation cell boundaries with larger size are η_p precipitates. The good combination of strength and ductility in the R20%-100 h

Fig. 6 Diagrammatic sketch of the forming of dislocation cells and precipitation morphology in the R20%-T6 and R80%-T6 sample upon aging treatment

sample is ascribed to the hierarchical dislocation and precipitate structure formed in the sample, i.e. small-sized GP- η' precipitates in the sub-grains and large-sized η_p precipitates on dislocation cell boundaries and residual dislocations. With increasing the degree of pre-deformation, the dislocation cell structure becomes difficult to be distinguished gradually, since the η_p precipitates distribute more and more homogeneous in the alloy.

2. The rapid nucleation and coarsening of the η_p precipitates and diminishing of the GP- η' precipitates in the alloy are responsible for the slight decrease in yield strength as the degree of pre-deformation increases from 20 to 80%.

Acknowledgements This work was financially supported by the National Natural Science Foundation of China (Nos. 51831004, 11427806, 51671082, 51471067, 11904093) and the National Key Research and Development Program of China (No. 2016YFB0300801).

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