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Microstructure and property of stress aged Al−Cu single crystal under various applied stresses

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Abstract: The stress aging behavior of Al−Cu alloy under various applied stresses, i.e., elastic stress, yield stress and plastic deformation stress, was investigated using single crystals. The resulting microstructures and the yield strength were examined by transmission electron microscopy (TEM) and compression tests, respectively. The results indicate that an elastic stress of 15 MPa is high enough to influence the precipitation distribution of θ' during aging at 180 °C. The applied stress loading along $[\bar{1}16]_{Al}$ direction results in increased number density of θ' on $(001)_{Al}$ habit planes. This result becomes more significant with increasing applied stress and leads to lower yield strength of Al−Cu single crystals during aging. Moreover, the generation of the preferential orientation of θ' was discussed by the effect of the dislocation induced by applied stress as well as the role of the misfit between the θ'-precipitate and Al matrix. The results are in agreement with the effect of the latter one.

Key words: Al alloy; single crystal; stress aging; θ' phase; microstructure; property

1 Introduction

Owning the superiority of high specific strength, excellent fatigue resistance and ease of formability, aluminum alloys are ideal materials to perform the creep age-forming technique that can finish the age-hardening treatment and stress-induced deformation simultaneously. This creep age-forming technique has been utilized in the manufacturing process of large integrally stiffened lightweight structures for aerospace applications [1−3]. However, application of an external stress during aging can strongly affect the precipitate distribution and generate preferential orientation of precipitates in several age-hardenable alloy systems. This preferential orientation of precipitates structure or aligned precipitate structure was usually called the stress-orienting effect.

The plate-shaped θ' phase, of nominal stoichiometry Al_2Cu, is one of the most common and effective strengthening precipitate phases in aluminum alloys [4], and provides significant contribution to the yield strength of parts of 2000 series aluminum alloys. The stress-orienting effect in Al alloy system was firstly reported in binary Al−Cu alloy [5], i.e the θ'-plate, which may be attributed to its special habit planes of $\{100\}_{Al}$ and elongation directions of $\langle 001\rangle_{Al}$. Thus, it was usually used to study the conditions of generating the stress-orienting effect [5−8], or the precipitation distribution structure induced by the stress aging [9−11]. Additionally, with the development of the creep age-forming technique, the researchers are more concerned with the effect of stress aging or creep aging behavior on the strength property [12−15]. It has been reported that the stress-orienting effect may reduce the yield strength and deteriorate the strength anisotropy [16,17]. However, what degree of the stress may affect the precipitation distribution of θ', and how it further affects the strength property are not clearly understood.

Furthermore, regarding the mechanism of the stress-orienting effect, LI [18] first explained it with classical nucleation theory in which external work by the applied stress compensates the strain energy due to the lattice misfit of coherent precipitate nuclei, and it was widely employed to explain the stress-orienting effect [5−9].

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However, since SANKARAN's explanation [19] took into account the preferred nucleation on the stress-oriented dislocation structures, there were also some studies proposed that the dislocation affected or even dominated the precipitation of the precipitates during stress aging [20,21]. In the present study, we described the results of our investigation on Al−2%Cu single crystal that was aged without stress and with various applied compressive stresses, i.e., elastic stress, yield stress and plastic deformation stress, which are determined by the stress−strain curve of the Al−Cu single crystal. The resulting microstructures and the yield strength, as well as the relationship between the microstructures and the strength property, were examined in detail. Based on the dispute about the explanations of the stress-orienting effect, both the effect of the dislocation induced by applied stress and the effect of the role of the misfit between the precipitate and the Al matrix on the precipitation of θ' were discussed.

2 Experimental

The material (Al−2%Cu) and the procedures (heat treatments and single crystal preparation) used for the present experiments were as described in a previous publication [22]. In order to apply different compressive stress magnitudes, the biggest one chosen from single crystals was cut into four parts with different dimensions. The orientations (rolled surface) of the single crystal were determined by an automated electron back-scattered diffraction (EBSD) system mounted in a FEI Nova 230 Nanolab scanning electron microscope (SEM), and performed at 20 kV and 10 mm scan steps. The preparation procedures of the samples for EBSD measurement and post-processing analysis of the orientation maps could refer to Ref. [15]. Analysis of the result showed that the plane orientations of the single crystals were $(\bar{1}16)_{Al}$. Additionally, one of these four single crystals was subjected to determine the stress−strain curve of the Al−Cu single crystal by compression tests using a strain rate 10^{-2} s^{-1} in a CSS44110 machine, and the other three single crystals were subjected to stress aging. One additional single crystal sample with random orientation was subjected to conventional aging (aging without stress) for microstructure comparison. These four single crystal samples were solution-treated at 525 °C for 2 h and quenched in water, and the stress-aging samples were loaded with compressive stresses in a specially designed fixture with three sample space, which was made according the Ref. [17]. Subsequently, all single crystal samples were aged at 180 °C for 66 h together. The applied stress magnitudes were approximately 15, 40, 60 MPa, respectively.

The yield stresses of aged samples were determined at 0.2% offset from the elastic response of compression tests on specimens using a strain rate 10^{-2} s^{-1} in a CSS44110 machine. The precipitate structure in the samples aged with and without stress was characterized by means of high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) and TEM. The TEM specimens were prepared by twin-jet polishing at 20 V using a solution of 50 mL HNO_3 and 150 mL methanol cooled at −25 to −30 °C. The TEM images and HAADF-STEM images were observed by FEI Titan G2 60-300 transmission electron microscope (TEM) operating at 300 kV. The features of the precipitates including the average diameter and the number density were determined by quantitative analysis of 5−10 TEM images for each sample.

3 Results

3.1 Stress−strain curve of Al−Cu single crystal before aging

As the stress aging samples of Al−Cu single crystal were subjected to applied stress prior to aging treatment, the applied stress may lead to a certain plastic deformation strain. In order to clearly understand the effect of the applied stress on aging behavior of Al−Cu single crystals during stress aging, it is necessary to determine the nature of the applied stress via the stress−strain curve of the Al−Cu single crystal. Figure 1 shows the stress−strain curve of the Al−Cu single crystal before aging (as-quenched condition). The resulting plastic deformation strain induced by corresponding applied stress are determined as shown in Table 1 based on Fig. 1. It can be found that the applied stresses during stress aging in present study are exactly located in different deformation stages, i.e., elastic stress ε_p=0.02% (15 MPa), close to yield point stress ε_p=0.3% (40 MPa), and plastic deformation stress ε_p=3.5% (60 MPa), respectively.

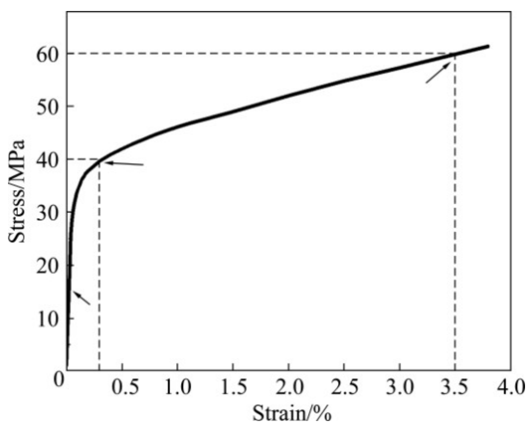

Fig. 1 Stress−strain curve of Al−Cu single crystal before aging (as-quenched condition)

Table 1 Various applied stresses during stress aging and corresponding resulting strain

Applied stress σ_a/MPa	Resulting strain ε_p/%
15	−0.02
40	−0.3
60	−3.5

3.2 Microstructure of Al−Cu single crystals stress aged under various applied stresses

Figures 2 and 3 depict the bright-field (BF) TEM images and HAADF-STEM images of Al−Cu single crystal samples aged under different applied stresses σ_a=0, 15, 40 and 60 MPa, respectively, loading along $[\bar{1}16]_{Al}$ direction for stress-aging samples. Using $[001]_{Al}$ zone axis of the electron beam in the TEM, the BF images show that two $\{100\}_{Al}$ variants of precipitates are visible in both stress-free (Fig. 2(a)) and stress aging samples (Figs. 2(b), (c)), even in larger applied stress aging samples with plastic deformation strain of 3.5% (Fig. 2(d)). Both the diffraction patterns and the dimension of plates indicate that most precipitates are plate-shaped θ'-phase. The aligned θ'-precipitate structure found in previous studies [6−9] can not be observed in the present study. This is due to the fact that two visible variants of θ'-phase in Fig. 2 are both nearly perpendicular to the loading orientation, while one variant of θ'-phase is perpendicular and the other one is parallel to the loading orientation in previous studies [9]. Actually, this result is consistent with the conclusion that compressive stress leads to a perpendicular θ'-plate dominated precipitate structure [9].

The mean diameter and number density of θ'-plates are quantitatively analyzed and summarized as shown in Table 2. It shows that the mean diameters of stress-aged samples are smaller as compared with the stress-free aged sample. Both Fig. 2 and Table 2 show that the number density of precipitates projected on the $(001)_{Al}$ habit plane is obviously increased when larger compressive stress is applied. This trend can be also confirmed by the observation of HAADF-STEM images in Fig. 3. Combining the results of previous studies proposed that larger applied compressive stress leads to more noticeable stress-orienting effect, it suggests that this increased number density in present study is another

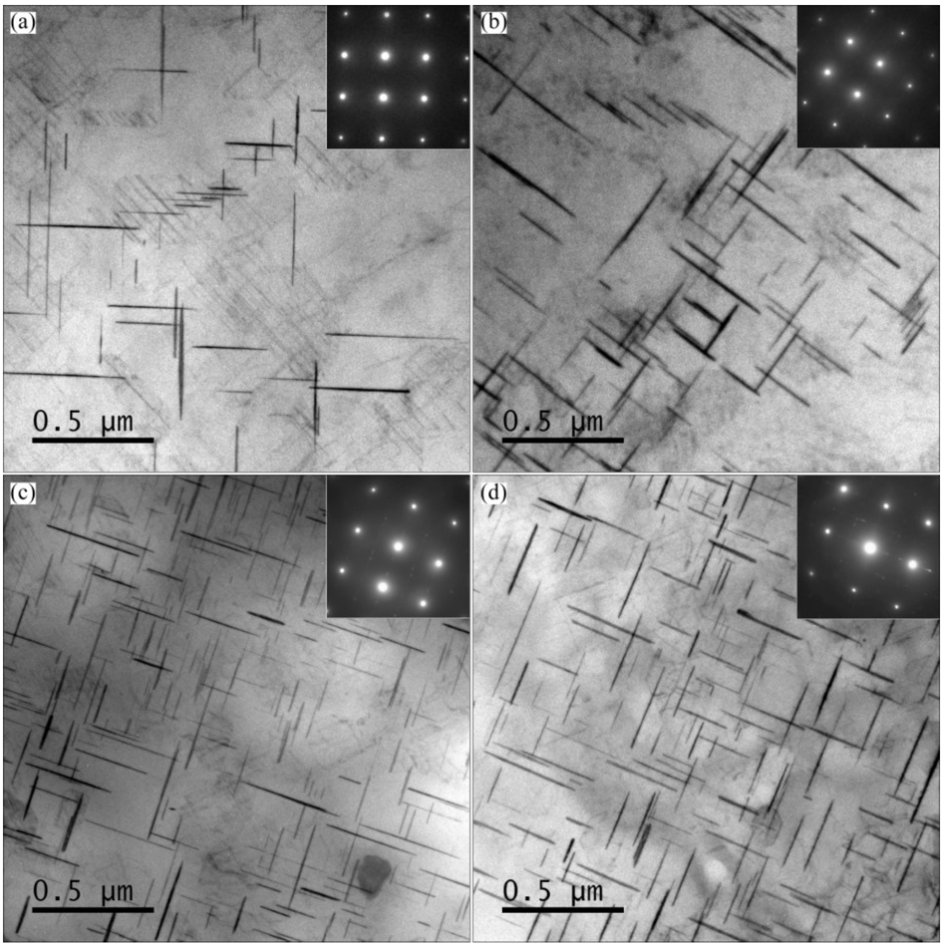

Fig. 2 BF TEM images and corresponding diffraction patterns of Al−Cu single crystals aged at 180 °C for 66 h under various compressive stresses: (a) 0 MPa; (b) 15 MPa; (c) 40 MPa; (d) 60 MPa (Compressive load orientation was along $[\bar{1}16]_{Al}$ for the stress aging samples)

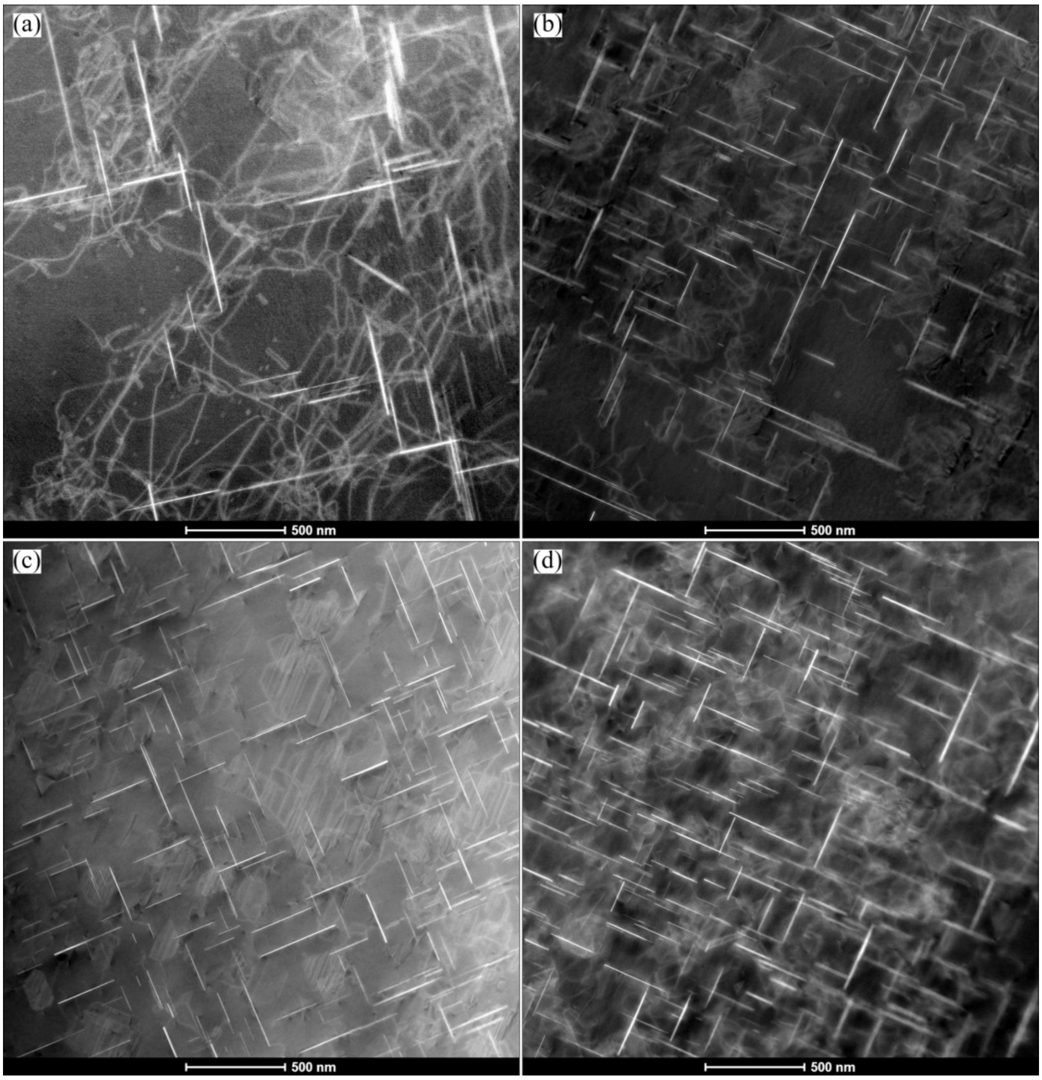

Fig. 3 HAADF-STEM images of Al−Cu single crystals aged at 180 °C for 66 h under various compressive stresses: (a) 0 MPa; (b) 15 MPa; (c) 40 MPa; (d) 60 MPa

Table 2 Mean diameter and number density of precipitates on $(001)_{Al}$ habit plane in Al−Cu single crystal samples under different applied stresses determined by quantitative analysis of TEM images

Applied stress/ MPa	Diameter (D)/ nm	Number density (N_v)/nm^3
0	307	0.16×10^{-7}
15	282	0.23×10^{-7}
40	238	0.45×10^{-7}
60	230	0.52×10^{-7}

sign indicating the stress-orienting effect. Thus, the results reveal that the elastic stress of 15 MPa is high enough to influence the precipitation of θ' during aging, and also indicate that larger applied stress resulted in more noticeable stress-orienting effect on θ' precipitation.

3.3 Yield strength of Al−Cu single crystals stress aged under various applied stresses

Figure 4 depicts the yield strength property of Al−Cu single crystals aged at 180 °C for 66 h under various applied compressive stresses loading along $[\bar{1}16]_{Al}$ direction, and the corresponding number density of θ' projected on $(001)_{Al}$. The data show that the yield stresses of stress-aged samples are lower than that of stress-free aged sample. It also shows that the yield stress of stress-aged sample exhibits decreasing tendency with increasing applied stress magnitude, and larger applied stress resulted in lower yield strength of Al−Cu single crystals during stress aging. Meanwhile, the change of the number density of θ' exhibits the exactly opposite tendency with that of the yield strength. Additionally, SKROTZKI et al [8] considered that the stress-orienting effect could not be generated unless a critical stress was exceeded, and proposed that the critical stress was between 16 and 19 MPa for θ' phase at the aging temperature of 160 °C. Based on the data of Fig. 4, the yield strength of the elastic stress-aging sample (15 MPa) is slightly lower than that of stress-free aged sample. We can therefore speculate that the critical stress for Al−2%Cu single crystal is lower, close to 15 MPa at the aging temperature of 180 °C. It is also supported by the slightly higher number density of θ' in the elastic stress aging sample as compared with that of stress-free aging sample (Table 2).

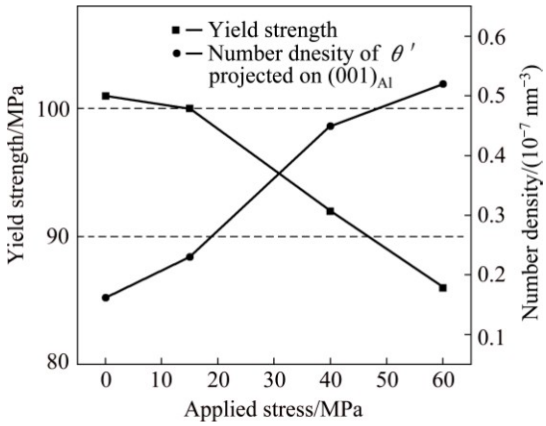

Fig. 4 Yield strength of Al−Cu single crystals aged at 180 °C for 66 h under different applied stresses, and corresponding number density of θ' projected on $(001)_{Al}$

4 Discussion

4.1 Effect of dislocations induced by applied stress on precipitation of θ' during stress aging

Generally, the number density of the dislocation induced by stress is depended on the resulting plastic deformation strain of the stress, and larder plastic deformation strain results in higher number density of the dislocations. Figure 5 shows the comparison for the microstructure of stress-free aged and stress-aged samples. It can be observed that the number density of the dislocations in the stress-aged sample at 60 MPa is significantly higher as compared with that of the stress-free aged sample. It can be therefore speculated that the number density of the dislocations in the stress aged sample at 60 MPa with deformation strain of 3.5% is also noticeably higher than that of the stress-aged sample at 40 MPa with deformation strain of 0.3% and the elastic stress aged sample (15 MPa). However, according to the result of Table 2, the number density of θ' in the stress aged sample at 60 MPa is only slightly higher as compared with that of the stress-aged sample at 40 MPa. On the contrast, the number density of θ' in the stress-aged sample at 40 MPa is significantly higher than that of the elastic stress aged sample at 15 MPa with the strain of 0.02%. Thus, it suggests that the effect of the dislocations induced by stress does not play crucial role on the precipitation distribution of θ'.

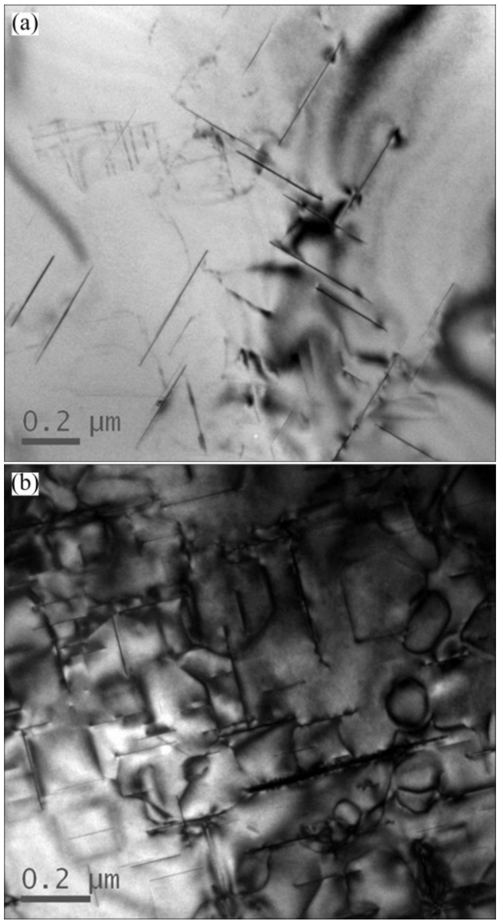

Fig. 5 Comparison for microstructure of samples aged under different stress: (a) Stress-free; (b) Plastic deformation stress (60 MPa) with deformation strain of 3.5%

4.2 Effect of role of misfit between θ' and Al matrix on precipitation of θ' during stress aging

The frame of classical nucleation and growth theories, which incorporate the interaction energy between the external stress and the strain fields due to the lattice misfits between the θ'-precipitates and the Al matrix, was also widely used for explaining the stress-orienting effect on θ' precipitation [6−9]. It was considered that the precipitation process of a coherent or semi-coherent second phase is affected by the elastic strain built up by the lattice misfits between the second phase particles and the matrix [9]. In the presence of an external uniaxial stress, the elastic strain may be either released or enhanced. Due to the interaction between the applied stress and the strain field, either an increment or decrement will be generated in the elastic strain energy associated with the formation the coherent particles. According to the analysis of this frame, a compressive stress loading along $\langle 001\rangle_{Al}$ direction releases the strain more for the perpendicular plates than for the parallel ones [7−9], due to a negative misfit of θ' phase ($\delta_{3,\theta'}$=−4.5% [8,9]). Therefore, the precipitation of perpendicular precipitates would be accelerated and more perpendicular θ' phase would precipitate, and this tendency would be more noticeable with increasing applied compressive stress, which is attributed to release more misfit strain. This may be more suitable to explain the increased number density of θ' with increasing applied stress observed in Fig. 2. It can be concluded that the results in the present study are in agreement with the effect of the role of the misfit between the precipitate and the Al matrix.

Additionally, based on the concomitant precipitation model [23,24], favorable perpendicular precipitates evolve at the expense of the unfavorable parallel ones throughout the whole precipitation process. Eventually, an aligned precipitate structure is formed in the habit planes parallel to the loading orientation, while a structure of two variants with increased number density is formed in the habit planes perpendicular to the loading orientation. This effect of stress aging on the precipitation of plate-shaped θ'-phase with increasing applied compressive stress could be simply drawn as Fig. 6. Thus, two different situations would be observed using different zone axis in the TEM images in the compressive stress aging condition: 1) The aligned θ'-phase precipitate structure would be observed if the zone axis ($[100]_{Al}$ or $[010]_{Al}$) is perpendicular to the loading orientation ($[001]_{Al}$); 2) Both of $\{001\}_{Al}$ variants are still visible if the zone axis ($[001]_{Al}$) is parallel to the loading orientation ($[001]_{Al}$), but the number density would be significantly increased. In other words, the increased number density can be also regarded as a symbol of the stress-orienting effect. Accordingly, we can understand why an aligned precipitates structure was found in stress aged Al−Cu alloy in previous studies [6−9], but two variants with increased number density was observed in the present study under similar stress aging condition. This may be also one of the reasons why some studies considered that the stress-orientation was not generated or was not obvious [12] while no aligned precipitate structure was found during stress aging.

4.3 Relationship between microstructure and yield strength of Al−Cu single crystals during stress aging

Strengthening precipitates were generally considered to provide the most significant contribution to the yield strength of age hardenable aluminum alloys [25]. According to the analysis mentioned above and Fig. 2, the applied stress during stress aging may deteriorate the uniformity of precipitation distribution of θ', i.e., increasing the number density on $(001)_{Al}$ while generating the preferential orientation of θ' on $(010)_{Al}$ and $(100)_{Al}$. This uneven precipitation distribution of θ' reduces the yield strength of the stress-aged samples as compared with the stress-free aged samples, which is consistent with the results of ZHU et al [17]. Furthermore, larger applied stress led to the increased number density of θ' on $(001)_{Al}$ but decreased yield strength based on the result of Fig. 4. Thus, it is reasonable to conclude that the yield strength of the single crystal is closely associated with the precipitation structure of the precipitates, and more uneven precipitation structure may further deteriorate the yield strength of the material during stress aging. On the other hand, the stress-aged sample at 60 MPa with the plastic deformation strain of 3.5% provided a relatively high number density of the dislocations (Fig. 5), which may inhibit the slip activity in the material and therefore enhance the yield strength of the sample. However, the yield strength of the stress aged sample at 60 MPa was still the lowest in all of the samples based on the data in Fig. 4. This reconfirms that the effect of the dislocations is far weaker than that of the precipitation distribution of θ' on the strength property, and this further suggests that the effect of a certain pre-deformation before stress aging is very limited on the precipitation distribution of θ' as well as the strength property for stress-aged Al−Cu alloy.

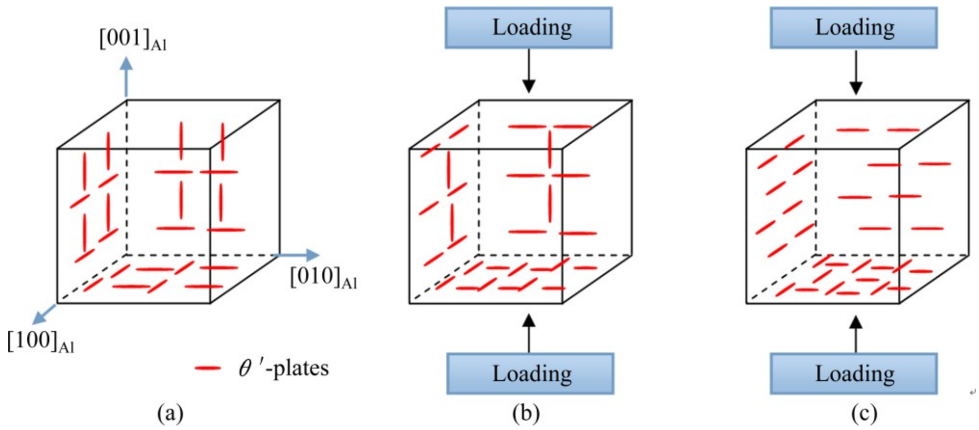

Fig. 6 Schematic diagram for effect of stress aging on precipitation of plate-shaped θ'-phase with increasing applied compressive stress magnitude: (a) 0 MPa; (b) 15 MPa; (c) 60 MPa

5 Conclusions

1) The elastic stress of 15 MPa was high enough to influence the precipitation distribution of θ' during aging, and larger applied stress loading along $[\bar{1}16]_{Al}$ direction resulted in increasing number density of θ' on $(001)_{Al}$ habit planes. This increased number density of θ' was also a sign of the stress-orienting effect.

2) The critical stress generated the stress-orienting effect on θ' precipitation was speculated to be lower, close to 15 MPa at the aging temperature of 180 °C.

3) The applied compressive stress reduced the yield strength of Al−Cu single crystals during aging, especially the yield stress and the plastic stress, which were attributed to their deteriorative precipitation distribution of θ'-$\{001\}_{Al}$ precipitates induced by corresponding stress aging.

4) The role of the misfit between the θ'-precipitate and Al matrix played crucial effect on the precipitation and the strength property of Al−Cu single crystals during stress aging, and the effect of the dislocation induced by applied stress was very limited.

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不同加载条件下 Al−Cu 合金单晶应力时效后的组织与性能

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摘 要：利用单晶研究了 Al−Cu 合金在弹性应力、屈服应力及塑性变形应力等加载条件下的应力时效行为，并分别通过透射电子显微镜(TEM)和压缩试验研究了应力时效后的显微组织与屈服强度。结果表明：15 MPa 的弹性应力就足够影响 Al−Cu 合金单晶在 180 °C 时效过程中 θ'相的析出分布。沿着 $[\bar{1}16]_{Al}$ 方向加载的应力会增加 Al−Cu 合金单晶 $(001)_{Al}$ 惯析面上 θ'相的析出密度。随着外加应力的增大，$(001)_{Al}$ 惯析面上 θ'相的析出密度则会随之增加，进而导致 Al−Cu 合金单晶的屈服强度降低。通过应力产生的位错的影响及 θ'相与 Al 基体之间的错配度作用的影响两个方面对应力时效时析出相择优分布的产生进行了讨论，结果更加符合 θ'相与 Al 基体之间错配度作用的影响。

关键词：Al 合金；单晶；应力时效；θ'相；显微组织；性能

(Edited by Yun-bin HE)