Effects of creep ageing on the mechanical properties and micro precipitates of X2A66 alloy

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Abstract: In the present work, effects of creep ageing on the mechanical properties and micro precipitates of X2A66 alloy are analysed herein. Compared with artificial ageing, creep ageing controls the formation process of fine T₁ nano precipitates. During CA treatment and thus facilitate the development of the mechanical properties of X2A66 alloy including its hardness and strength. However, the elongation of the alloy is slightly reduced.

1. Introduction
Li is the lightest metal in nature, and each 1% (mass fraction) boost of Li content in aluminum alloy contributes to about 3% density reduction and about 5%-6% elastic modulus increase. Aluminum-Lithium alloys have been successfully applied in the aeronautic and astronautic industry [1], such as Airbus 350/380, Boeing 777 and C919 aircraft. China has developed a fourth-generation Al-Li alloy (X2A66) with independent intellectual property rights by adjusting the contents of Cu and Li and adding other micro-alloying elements (such as Mg, Zn, Zr, Mn) in Al-Li alloys. X2A66 alloy is beneficial to realize material self-weight reduction and structural design weight loss.

Due to the lightweight and promising properties, the third generation Al-Cu-Li alloys are used in manufacturing rocket tanks and airplane wings/body [2]. Through creep ageing (CA) treatment, these alloys exhibit excellent strength and sounds fatigue resistance. Based on CA, a new approach, namely creep age forming, was recently developed [3]. By combining the creep and artificial ageing (AA) processes together, Creep age forming can form aluminium alloy sheets within its elastic limit and strengthen the material simultaneously, making it a particularly appropriate forming technology for large panels in the aerospace industry. Libin Hu et al. [4] studied the effect of creep ageing on the mechanical properties and micro precipitates of Al-Li-S4 alloy. Their results indicate that CA has significantly improved the mechanical properties of Al-Li-S4 alloy. It has been predicted that mechanical properties can be significantly enhanced using such an approach. Many researchers [3-5] have studied the creep aging behavior of Al-Li alloys, but the creep aging behavior of X2A66 Al-Li alloy has not been reported yet. The aim of this work is to reveal the effect of CA on the mechanical properties and micro precipitates of X2A66 alloy.
2. Material and experimental procedure

In this paper, X2A66 alloys were laboratory-made. Binary intermediate alloys of Al-10Li, Al-50Cu, Al-10Zr and Al-10Mn and pure elements of Mg, Zn and high purity ingot of Al were melted in a vacuum induction melting furnace under a controlled atmosphere of argon gas, using high pure graphite crucible. Using SG-XS highly temperature box typed to muffle furnace to homogenize the ingot. Samples are hot-rolled at 450°C in multiple passes to 4mm and then cold-rolled to 2mm in thickness by the Φ400*1000 two-roll hot rolling mills. The chemical composition of the X2A66 alloy examined in this investigation is shown in Table 1.

The samples were machined out from the cold-rolled plate. Samples’ longitudinal direction was parallel to the rolling direction. CA and AA samples were prepared as a standard sample to investigate the features. The AA test sample was given a solution heat treatment (1h at 510°C), then water quenched and followed artificially aging (175°C) for the DHG9030 digital display thermostatic dryer (temperature error±3°C). Artificial aging time is 0, 4, 8, 12, 16, 20, 24, 28 and 32h respectively. After solid solution, in order to reduce the effect of natural aging at room temperature, the CA test is started within one hour. The CA (under a tensile stress of 200MPa) was carried out in a furnace at 175°C on the RDL50 electronic creep relaxation test machine for 4, 8, 12, 16, 20, 24, 28 and 32h, respectively. During CA tests, the exterior stress was smoothly loaded at a rate of 75N/s using computer-aided controlling system. After the sample is cooled to room temperature in air, samples are taken for mechanical properties testing and microstructure analysis.

Vickers micro-hardness (HV, Wilson TuKon 1202) was measured to evaluate the strengthening behavior. A load of 100g was applied for a holding time of 15s. At least 5 points were measured for each sample. Mechanical tensile testing was carried out using a DDL100 tensile machine at a strain rate of 2mm/min at room temperature. The foils for transmission electron microscopy (TEM, FEI TECNAI G² 20) observations were prepared by mechanical polishing to less than 70μm, subsequently punched into 3mm disks and final twin-jet polishing with an electrolyte solution of 20% nitric acid and 80% methanol at the voltage of 10–20V DC and the temperature below −20°C.

Table 1 Chemical compositions of the X2A66 alloy (wt.%)

| Alloys | Cu  | Li  | Mg  | Mn  | Zn   | Zr   | Ti  | Fe  | Si  | Al  |
|--------|-----|-----|-----|-----|------|------|-----|-----|-----|-----|
| X2A66  | 3.5~4.1 | 1.3~1.8 | 0.2~0.6 | 0.2~0.6 | 0.2~0.8 | 0.08~0.16 | <0.1 | <0.1 | <0.1 | Bal. |

3. Results and discussions

3.1 Enhanced hardening from artificial ageing

In Fig. 1, the variation of the average hardness of X2A66 alloy with different ageing time is plotted. From Fig. 1, it can be seen that the alloy have a significant age hardening effect. As the aging time increases, the hardness of the alloy gradually increases until the hardness reaches a plateau at 24h. The peak hardness is 187.14HV. It can further be observed from Fig. 1 that the peak hardness of all the samples exhibits a maximum plateau region that remains stable when they are artificially aged for up to 60h.
3.2 Mechanical properties after aging

Fig. 2 presents the tensile response of X2A66 alloy under different ageing treatments. As can be observed in Fig. 2(a), the first feature of interest is that the yield strength (YS) / ultimate tensile strength (UTS) of the specimens prepared by AA approach is always higher than CA before 32h. The AA samples peaked at 24h, and the yield and tensile strength were 556.11 and 593.16MPa, respectively. Then, they slightly decreased to 515.68 and 576.16MPa at 32h. The strength test results are consistent with the hardness test results in Fig. 1. However, YS/UTS of CA-treated specimens continuously increased from initial 260.25/360.78MPa at 0h to 534.82/578.16MPa at 32h. The elongation of specimens prepared either using AA or CA was monotonically reduced along the ageing time (see Fig. 2(b)) and the elongation value of the samples after AA treatment is always higher than CA.

Experimental parameters are closely associated with the precipitation behavior of the Al-Cu-Li alloy. YS and UTS depend on the combined contribution from multiphase precipitates. \( T_1(\text{Al}_2\text{CuLi}) \), \( \delta'(\text{Al}_3\text{Li}) \) and \( \theta'(\text{Al}_2\text{Cu}) \) are the main strengthening phases in the Al-Cu-Li alloys, of which the \( T_1 \) nano precipitates were recognized to make a major contribution to hardening alloys.

Thus, the time to reach the peak strength of the alloy may vary in the specimens with different constituents (or volume fraction) of multiphase precipitates. The maximum volume fraction of \( T_1 \) nano precipitates was obtained beyond the general peak-ageing time. In other words, CA prolonged peak-ageing time than AA. Statistical analysis of \( T_1 \) nano precipitates will be given in Section 3.3.

3.3 micro precipitates after aging

TEM images and the selected area diffraction (SAD) patterns of the X2A66 alloys treated using different approaches are shown in Fig. 3. All TEM images and SAD patterns were taken close to a [110] Matrix.
axis. TEM dark field (DF) images and SAD patterns of AA-treated specimens were shown in Fig. 3(a) and (b). The samples in Fig. 3(a) and (b) corresponded to the X2A66 alloys which were aged at 175°C for 24h. TEM DF images and SAD patterns of CA-treated specimens were shown in Fig. 3(c) and (d). The specimens corresponding to Fig. 3(c) and (d) were prepared using the CA approach at 175°C for 32h, where an exterior stress of 200MPa was applied. According to Refs. [6], the SAD patterns in Fig. 3(b) and (d) displayed the presence of δ', θ' and T1. Superlattice points (at the center of rhombus diagonal) demonstrate the existence of δ'. Indexed {200} Matrix diffraction points verifies the existence of θ' phase along [110] Matrix axis. Diffraction spots, positioned at 1/3 and 2/3 of the distance from {000}Matrix to {02-2}Matrix spot, verifies the existence of T1 precipitates. Points with stripe crossing (11-1)Matrix and (1-11)Matrix indicate that T1 precipitate is following the direction of [11-1]+[1-11] with a certain angle. The diffraction spot of T1 is stronger compared with θ' and δ'. It is known from Fig. 3(b) and (d), compared with AA, the θ' and δ' spots are all weaker than CA. And CA samples generated a remarkable increase in the fine T1 precipitates, as showed in Fig. 3(a) and (c).

P. Donnadieu etal. [7] results show that dislocations can provide the extra nucleation sites for T1 precipitates. However, formation of T1 precipitates occupies lots of Li and Cu atoms from Al matrix, which results in decreasing the number of δ' and θ' phases. Under artificial ageing, the multiphase precipitates include δ', θ' and T1. In CA, dislocations are unavoidable. Through initiating favourable dislocations, the exterior stress during CA treatment will promote the nucleation of T1 nano precipitates. As a result, the critical time for reaching peak ageing was prolonged and the precipitation of δ' and θ' phases is suppressed. The theoretical analysis is consistent with the experimental results of Fig. 2 and 3.

Fig. 3(e) and (f) presents the micrographs of the precipitates at the grain boundary and the precipitate free zone (PFZ) with artificial aging time of 24h and creep aging time of 32h, respectively. Formation of PFZ is related to the granular shortage zone at the neighboring grain boundaries. The inter crystalline fracture in an alloy is mainly attributed to the precipitates at the grain boundary and PFZ. As can be seen from Fig 3(e), at the grain boundary, the mixed precipitates contain δ', θ' and T1. Furthermore, a certain width of PFZ is formed. As showed in Fig. 3(f), compared with AA, the CA-treated sample produced a wider PFZ and precipitated a coarser precipitated phase at the grain boundaries. In the tensile test, the alloy occurs plastic deformation under the axial tensile stress, and deformation precedence over the PFZ will cause the stress set in this region. Micro cracks and micropores will be extended and coarsened along the PFZ, and the degradation of the alloy leads to the deterioration of the toughness of the alloy and causes the low energy inter granular fracture of the material. So the AA treatment sample has better toughness than CA.
Fig. 3 The TEM images of the AA-treated sample aged at 175°C for 24h: (a) DF, (b) corresponding SAD patterns and (e) Bright field TEM, The TEM images of the CA-treated sample aged at 175°C for 32h: (c) DF, (d) corresponding SAD patterns and (f) Bright field TEM.
Fig. 4 Fracture surfaces after tensile testing: (a) the AA-treated sample aged at 175°C for 24h, (b) the CA-treated sample aged at 175°C for 32h

The fractographic images in Fig. 4 identify certain dimples on each specimen, which shows that the major ductile fracture exhibited in all these samples. For AA sample, more fine dimples appeared on the fracture surface (Fig. 4(a)), indicating more ductile fracture mode. For CA sample, ductile fracture was less obvious with evidence of mixed dimples and tearing ridges distributed on the fracture surface (Fig. 4(b)). Normally, a large number of fine dimples imply sound ductility, and then large elongation. This is consistent with the experimental results shown in Fig. 2(b).

4. Conclusions

Compared with AA treatment, CA treatment initiates favourable dislocations and the exterior stress during CA treatment will promote the nucleation of T1 nano precipitates. As a result, the critical time for reaching peak ageing was prolonged and the precipitation of δ' and θ' phases is suppressed. Using CA treatment, the hardness and strength of X2A66 alloy were improved. Compared with AA-treated samples, the CA-treated sample produced a wider PFZ and precipitated a coarser precipitated phase at the grain boundaries. Boundaries, which leads to the deterioration of the toughness of the alloy.

Acknowledgements

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