



Enhanced fatigue crack propagation resistance in a superhigh strength Al–Zn–Mg–Cu alloy by modifying RRA treatment

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ABSTRACT

A new retrogression and re-aging (RRA) treatment was designed to enhance the fatigue crack propagation (FCP) resistance of a superhigh strength Al–Zn–Mg–Cu alloy in this work. As compared to traditional RRA treatments, a lower retrogression temperature and a longer retrogression and re-aging time were employed. This modified RRA processing obtained a coarse precipitates in matrix, and a slightly increased precipitate free zone (PFZ) width at grain boundary. This led to an almost complete transgranular crack propagation, rather than a partial intergranular crack propagation as traditional RRA tempered samples. This kind of fatigue crack propagation manner gave rise to an ultralow FCP rate, comparable to fatigue resistant 2000 series aluminum alloy, in an Al–Zn–Mg–Cu alloy while keeping superhigh tension strength.

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1. Introduction

Al–Zn–Mg–Cu series aluminum alloys have draw much attentions as an important aeronautical material due to the combination of good mechanical property and low density [1–3]. However, the alloys in T6 condition represented a serious susceptibility to the stress corrosion cracking (SCC). The over-aging heat treatment could remedy this deficiency, but this approach resulted in a sacrifice of mechanical strength [4,5]. RRA treatment proposed by Cina [6] has been applied to the Al–Zn–Mg–Cu series alloys in order to improve the performance of the SCC resistance. This heat treatment involves three steps including pre-aging treatment, retrogressing by heating to a relatively high temperature for a short time, and re-aging at relatively low temperature similar to the pre-aging treatment. It has been reported that during retrogression, G.P. zones and fine η' particles are dissolved in matrix, and isolated grain boundary particles are formed, while in the re-aging stage, η' particles are re-precipitated [7,8]. This characteristic microstructure leads to a good combination of the mechanical performance and SCC resistance [9,10,11].

Recently, more investigations focused on the fatigue crack initiation and propagation behavior of the aluminum alloys [12,13]. The results indicated that the factors influencing the fatigue resistance should include grain size, grain orientation, grain boundary microstructure, dislocation configurations, secondary phase and inclusion particles [12,13].

For the Al–Zn–Mg–Cu alloys, precipitates in grain interior and grain boundaries remarkably influenced the fatigue fracture behavior [9,14,15]. Chen [9] proposed that in Al–Zn–Mg–Cu alloy, RRA-tempered samples represented a lower FCP rate than T761-tempered samples, owing to the shearable grain interior precipitates and relatively narrow PFZs. In T761-tempered condition, intergranular crack path was found to predominate fatigue crack propagation in Paris regime. In contrast, both transgranular crack path and intergranular crack path were present in the RRA-tempered samples [9]. Chen [15] also revealed wide and soft PFZs in over-aged samples acted as a preferred route for intergranular crack propagation. This suggested the wide and soft PFZs degraded FCP resistance. Therefore, it is necessary to reduce the intergranular crack propagation for the sake of enhancing FCP resistance. Although the RRA treatment was confirmed to be beneficial to transgranular crack propagation by narrowing PFZs, but a complete transgranular path for FCP at high ΔK levels was never found in the RRA-tempered alloys. In order to avoid the intergranular crack propagation, besides strengthening grain boundaries, it is well understandable that appropriately decreasing the strength of grain interiors could be another practicable approach. Decreasing the strength of grain interiors could be achieved by enlarging the precipitate size in the matrix. Evidently prolonging re-aging time during RRA processing, is able to enlarge both the precipitate size and spacing. An enlarged precipitate spacing, in no doubt, increases the free slipping distance of dislocations. This is beneficial to the reversible dislocation slipping at fatigue crack tip and crack closure, and finally reducing fatigue damage accumulation.

In our previous work [9], by means of RRA treatment, an Al–Zn–Mg–Cu alloy with low zinc content was offered high fatigue resistance. This

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alloy shows a relatively low strength as compared to the Al–Zn–Mg–Cu alloy with high zinc content. For the latter alloy, high zinc content bring about more and denser precipitates. Consequently this alloy represents an ultrahigh strength but a relatively worse plasticity. Meanwhile, dense secondary particles restrict the reversible slip of dislocations, resulting in a decrease of fatigue resistance. Besides, narrowing the PFZ width is another key issue. The PFZs width is sensitive to the retrogression temperature, and a low retrogressing temperature will slow the growth of GBPs through absorbing surrounding solute, thus leading to the formation of narrow PFZs. In traditional RRA treatments, the retrogressing temperature is commonly at around 200 °C [9,16,17]. Therefore, it is well understandable to select an appropriately lower retrogression temperature to reduce the PFZs width. Based on the above, a modified RRA process was designed for the high-zinc Al–Zn–Mg–Cu alloy to enhance FCP resistance while keeping ultrahigh tensile strength in this work. This modified RRA process aims at avoiding intergranular FCP. On the one hand, a low retrogression temperature was employed to restrict the overly wide PFZs, sequentially increasing the deformation resistance of grain boundaries. On the other hand, the retrogression and re-aging were prolonged to coarsen grain interior precipitates, so as to decrease the grain interior strength, thus the crack preferring to propagate into the grain interiors.

2. Experimental procedures

The composition of the materials used in the present investigation is Al–8.1Zn–1.7Mg–2.2Cu–0.15Zr (wt%). The alloy was received in homogenized condition, hot rolled to 3 mm in thickness. The sheet materials were, then subjected to a two-stage solution (450 °C for 2 h followed by 470 °C for 1 h), quenched in water. Then the sheet materials were classified to two group samples, which were respectively subjected to different RRA treatments shown in Table 1. In contrast with traditional RRA treatment, in which retrogression temperature adopted, was normally from 190 °C to 200 °C, a lower retrogression temperature (160 °C) was employed in this investigation. This retrogression temperature was determined by the result of the differential scanning calorimetry (DSC) analysis, as shown in Fig. 1. The first endothermic peak starting at 139.3 °C and reaching the peak at 168.7 °C indicated the dissolution of η' precipitates. In this investigation, employing a relatively low retrogression temperature aimed at avoiding excessively wide PFZs. Tensile testing was conducted on a CSS-44,100 tension machine with 2 mm/min loading speed. The sample for tension testing was prepared in long transverse (LT) direction of the sheet. Microstructural observations and corresponding selected area electron diffraction (SAED) analysis in both conditions were conducted by a Tecnai G²20 transmission electron microscopy (TEM) with an operating voltage of 200 kV. The compact tension (CT) samples of 45.6 mm × 38 mm × 2 mm (L × W × B) in size, taking from the sheet in LT orientation, were prepared for FCP test. The FCP testing was performed on an MTS machine at room temperature and in air. And a sinusoidal cyclic constant loading with a stress ratio ($R = \sigma_{\min}/\sigma_{\max}$) of 0.1 and a frequency of 10 Hz was applied in FCP test. Fatigue fractures were observed by a FEI Quanta 200 scanning electron microscope (SEM) with an operating voltage of 15 kV. EBSD samples were prepared by mechanical grind, and electropolishing in a solution of 90% ethanol and 10% perchloric acid. All EBSD experiments were performed on a FEI Helios Nanolab 600i field emission gun scanning electron microscope with an accelerating voltage of 20 kV.

Table 1

The parameters of two different RRA treatments.

Condition	Pre-aging	Retrogression	Re-aging
RRA1	100 °C/24 h	160 °C/10 min	100 °C/24 h
RRA2	100 °C/24 h	160 °C/120 min	100 °C/96 h

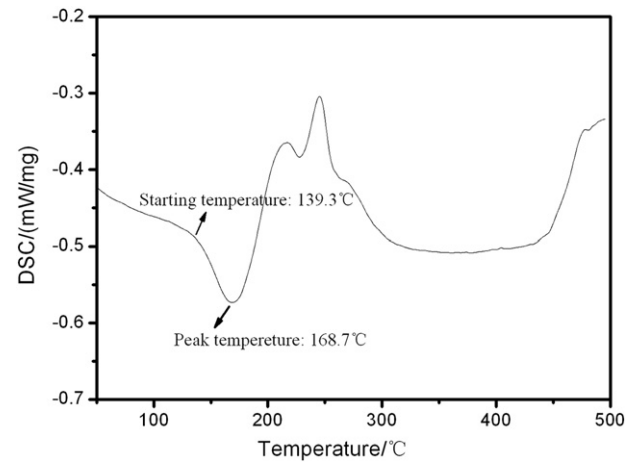


Fig. 1. DSC scan carried out on the Al–Zn–Mg–Cu alloy in T6 condition.

3. Results

3.1. Tensile properties

The tensile properties of the two conditions were represented in Table 2. It could be found that the RRA1-processed sample had relatively higher tensile and yield strengths as compared to the RRA2-processed sample, but both of them had equally matched elongations.

3.2. Fatigue crack growth

Fig. 2 illustrated the fatigue crack propagation rate curves (da/dN versus ΔK) of the samples treated by RRA1 and RRA2 treatments. Results showed RRA2-processed sample had a lower fatigue crack propagation rate and a relatively higher threshold value (ΔK_{th}) than RRA1-tempered sample. For instance, at $\Delta K = 10 \text{ MPa}\sqrt{\text{m}}$, the FCP rate of RRA2-processed sample reached $1.71 \times 10^{-4} \text{ mm/cycle}$, much lower than RRA1-processed sample ($3.42 \times 10^{-4} \text{ mm/cycle}$). The eventual fracture occurred at ΔK of 29.7 $\text{MPa}\sqrt{\text{m}}$ for RRA1-tempered sample, with a FCP rate of $2.23 \times 10^{-3} \text{ mm/cycle}$. In contrast, for RRA2-tempered sample, the eventual fracture occurred at ΔK of 32.5 $\text{MPa}\sqrt{\text{m}}$ with a FCP rate of $2.39 \times 10^{-3} \text{ mm/cycle}$. This ΔK value is markedly higher than that of the low-zinc Al–Zn–Mg–Cu alloy (about 28 $\text{MPa}\sqrt{\text{m}}$ while the FCP rate reached $3.94 \times 10^{-3} \text{ mm/cycle}$) referred to the previous work [9]. Meanwhile, this low FCP rate in RRA2-tempered sample is nearly equivalent to that in 2000 series aluminum alloys mentioned in the Ref. [18–20] at the same ΔK . Evidently, RRA2-processing approach, remarkably reducing FCP rates on the ground of ultrahigh tensile strength, was significant for raising damage tolerance of aeronautic aluminum alloys.

3.3. Microstructural characterization

As shown in Fig. 3(a), dense and fine precipitates in grain interior exhibited in RRA1 sample. In the SAED pattern showed in Fig. 3(a), the bright diffraction spots at 1/3 and 2/3 of $\{220\}_{\text{Al}}$ suggested the existence of η' precipitates, while the slightly weak diffraction spots at $\{1, 3/4, 0\}_{\text{Al}}$ in $\langle 100 \rangle$ Al projection corresponded to a small quantity of GPI zones [21]. It meant that the grain interior precipitates in RRA1-tempered sample mainly consisted of η' precipitates and GPI zones. In contrast,

Table 2

The tensile properties of RRA-treated Al–Zn–Mg–Cu alloy.

Condition	Tensile strength(Mpa)	Yield strength(Mpa)	Elongation(%)
RRA1	630	578	13.9
RRA2	613	551	13.6

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