Materials and Design 101 (2016) 16–23

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Aging behavior and precipitate characterization of a high Zn-containing Al-Zn-Mg-Cu alloy with various tempers

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ARTICLE INFO

Article history:
Received 13 January 2016
Received in revised form 14 March 2016
Accepted 30 March 2016
Available online 2 April 2016

Keywords:
Al-Zn-Mg-Cu alloy
Aging treatment
Precipitate
Mechanical properties

ABSTRACT

In the present work, the influence of one-step and two-step aging treatments on hardness, electrical conductivity and mechanical properties of a high Zn-containing Al-Zn-Mg-Cu alloy is investigated and detailed aging parameters subjected to various aging tempers, i.e., T6, T79, T76, T74 and T73, are proposed. The nanoscale precipitates under different tempers are qualitatively investigated by means of transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HREM) techniques. Based on the precipitate observations, precipitate size distributions and neighbor precipitates distances are extracted from bright-field TEM images projected along $\langle 110 \rangle_{Al}$ orientation with the aid of an imaging analysis. The results show that with the deepening of aging degree, the conductivity of one-step and two-step aging increase continuously while the hardness increases for one-step aging and decreases for two-step aging at the second preservation. The tensile strength decreases as the aging degree deepens and the yield strength shows a similar trend. In addition, as the degree of over-aging deepens, the precipitate size distribution interval becomes broader, the average precipitate size turns larger and the average distance of neighbor precipitates also becomes greater. The influence of precipitates on mechanical properties is discussed.

1. Introduction

The Al-Zn-Mg-Cu (7xxx series) aluminum alloys are extensively used in the commercial aircraft structures as well as various critical military bridges and vehicles due to their excellent mechanical properties, high strength to weight ratio, fracture toughness and SCC resistance [1–3]. In the past decades, 7xxx series aluminum alloys and their process innovation have led to higher strength-level than before, with higher content designing of the major alloying elements (mainly the zinc element) [4]. Based on industrial application requirements, different aging tempers have been developed for the 7xxx series, such as T6, T7X and RRA treatments [5]. After the single-stage aging (T6), the Al alloy shows a high strength but low corrosion resistance [6]. Two-stage aging (T7X) ensures an improvement in fracture toughness and stress corrosion resistance but a 10–15% reduction in strength compared with that at T6 temper [7,8]. In order to obtain comprehensive performance, the RRA treatment is developed whose strength is equal to that after T6 aging and stress corrosion resistance is close to that after T7X treatments [9,10]. However, the disadvantages of complex process and long aging time (usually more than 48 h) restricted its application. Hence, good aging parameters used for various tempers are important to application performance.

Universally, different aging treatments result in different microstructure characteristics (mainly refer to precipitates geometrical characteristics and their mismatch relationship with the matrix) and thus generate different performances to the alloy. It is the precipitation which provides by far the strongest contribution to the strength of aged Al-Zn-Mg-Cu alloys [11–13].During aging, the usual precipitation sequence which dominates strengthening in most commercially used 7xxx alloys can be summarized as follows [4,10,14–17]: SSSα → GP zones → η′ → η. Hence, quantitative information on nanoscale precipitates can be significantly helpful for evaluating alloy performance and the design of aging tempers.

The previous study of 7xxx series aluminum alloys has focused on application fields. Namely, the influence of aging treatments on various properties, like mechanical properties, stress corrosion resistance, etc. Meanwhile, the transmission electron microscopy (TEM), as a conventional and fairly applicative tool, has been extensively used on qualitative analysis on nanoscale precipitates in Al-Zn-Mg-Cu alloys [10,16, 18–20]. The zinc content of those investigated alloys is not high, most of them has a zinc content of no more than 9 wt.%. The Al-Zn-Mg-Cu alloys with very high Zn content higher than 9 wt.% possess higher strength and improved overall performance aims to replace conventional alloy for aerospace applications. The current literatures which

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http://dx.doi.org/10.1016/j.matdes.2016.03.150
0264-1275/

investigate Al-Zn-Mg-Cu aluminum alloys with high Zn-containing (more than 9 wt.%) have paid more attention to annealing processes while the study of aging behavior is not much and quantitative analysis on precipitates under various tempers is still very limited.

Generally speaking, the TEM makes it possible to characterize the precipitate size distribution by using an image analysis, but also neighbor precipitate distance simultaneously to the image analysis of the precipitates present [21]. Moreover, the scale of TEM analysis is hundreds of precipitates, which shows a fine and reliable statistical result. Hence, it is appropriate to use TEM images to quantitatively analyze precipitates.

In the present work, the evolution of hardness, electrical conductivity and mechanical properties in a novel high Zn-containing Al-Zn-Mg-Cu alloy (Zn content is close to 9.8 wt.%) during one-step and two-step aging treatments has been investigated and typical aging treatment parameters subjected to different aging tempers (including T6, T79, T76, T74 and T73) are proposed. The qualitative study of precipitates under those tempers is carried out by the transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HREM) techniques. The Bright field TEM images projected along $\langle 110\rangle_{Al}$ orientation are used to quantitatively analyze the geometrical characteristics of nanoscale precipitate. The influence of precipitates on mechanical properties is discussed.

2. Experimental procedure

The investigations were carried out on specimens cutting from a extruded Al-Zn-Mg-Cu alloy plate with chemical composition of Al-9.78Zn-2.04Mg-1.76Cu-0.11Zr-0.056Fe (in wt.%). The specimens were solution heat treated at 475 °C for 4 h, cold water quenching, followed by the typical one-step and two-step aging treatments. According to previous research work, 120 °C was chosen as the one-step aging temperature and the two-step schedule was firstly aged at 110 °C for 8 h and then preserved at 160 °C. The aging hardening processes were monitored by a 430SVD Vickers hardness tester using a loading force of 5 kgf and a dwell time of 15 s. Each hardness datum of the samples was the mean value of ten indentations. The electrical conductivity of the specimens was measured using the WD-Z eddy current meter at room temperature and owns the same datum collecting techniques as the hardness. Tensile specimens were prepared following specifications in ASTM standard E 517-00 with the gauge length parallel to the rolling direction. The tensile properties of the alloy under various aging tempers were measured by a WD3100 test machine at a constant speed of 2 mm/min. The TEM examinations were conducted on a JEM-2010FX transmission electron microscope, operating at 200 kV. Three millimeter diameter disks for TEM observation were punched out directly from slices which were mechanically ground down to 50 μm thickness. These disks were electro polished using a twinjet machine with a 25% nitric acid solution in methanol at −30 °C and 15–25 V. The detailed methods for measuring the precipitate size distribution and neighbor precipitate distance were given in Section 3.3.

3. Results and discussion

3.1. Hardness, conductivity and mechanical properties

The hardness and electrical conductivity curves of the investigated alloy aged at 120 °C are shown in Fig. 1(a). The alloy shows a rapid increase in hardness during the first 2 h, followed by a more gentle increase for up to 96 h. The hardness value of the alloy aged for 0.5 h is 188.2 HV. The value increases to 201.9 HV aged for 2 h, which shows an incremental percentage of 7.3% compared with the value of 188.2 HV. The hardness value increases to 210.4 HV aged for 24 h and the incremental percentage is 11.8%, which shows an augment of 8.5 HV in comparison to the value aged for 2 h. After this the increase is very slow and the hardness value reaches 214.8 HV. It means the augment from 24 h to 96 h is only 4.4 HV. Correspondingly, the electrical conductivity of the alloy aged at 0.5 h, 2 h, 24 h, and 96 h are 16.4 $MS\cdot m^{-1}$, 16.6 $MS\cdot m^{-1}$, 17.6 $MS\cdot m^{-1}$ and 18.8 $MS\cdot m^{-1}$, respectively. It indicates that the increment of conductivity after aged for 24 h slows down, which is consistent with the hardness curve. This demonstrates that the one-step aging treatment gets close to the peak precipitation strengthening state around 24 h and prolonged heat preservation makes no significant enhancement on the strengthening.

Fig. 1(b) shows the hardness and electrical conductivity curves of the two-step aging treatments. The hardness curve manifests a relatively homogeneous decrement for different preservations at 160 °C while the rise of electrical conductivity is also near uniform. Both have no obvious rapid change. This indicates that the degree of over-aging deepens with preservation time.

According to the hardness and electrical conductivity curves, samples treated by some specific aging conditions are chosen to test the room temperature tensile properties and the corresponding curves are presented in Fig. 2(a) for one-step aging and Fig. 2(b) for two-step aging treatments. As shown in Fig. 2(a), the ultimate tensile strength (UTS) has no obvious change with aging time while the yield strength (YS) curve reaches a peak and then maintains nearly at a plateau though there are small fluctuations. Hence, aging at 120 °C for 24 h can be treated as the T6 temper and the ultimate tensile strength and yield strength are 701 MPa and 654 MPa, respectively. This gives a reference to confirm the strength values of the T7X tempers. Fig. 2(b) shows the ultimate tensile strength and yield strength decrease with prolonging preservation time, which is similar to the hardness curve. Comprehensively considering the extents of strength decrement and conductivity increment [as shown in Fig. 1(b)], the preservation of 5 h, 9 h, 14 h and 24 h at 160 °C are chosen as suitable T79, T76, T74 and T73 tempers.

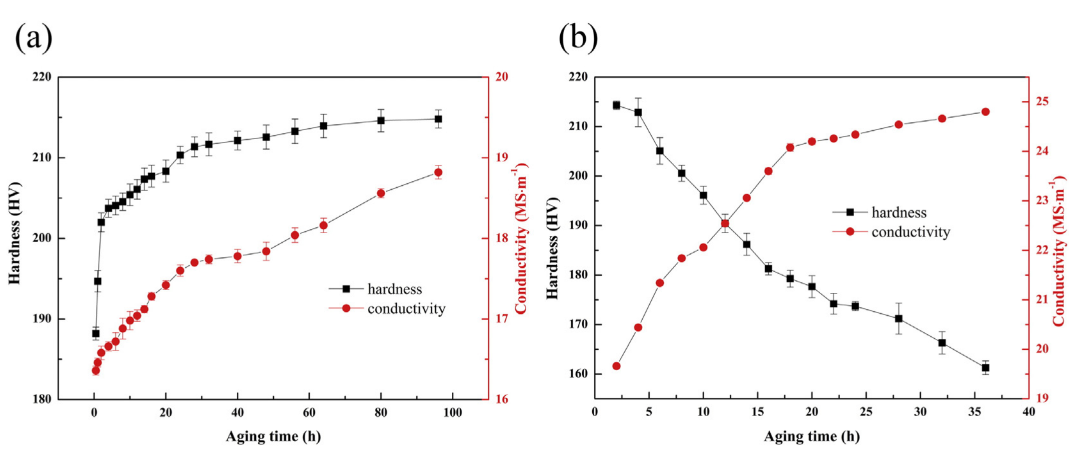

Fig. 1. The hardness and electrical conductivity curves of the alloy (a) aged at 120 °C and (b) aged 110 °C for 8 h and then preserved at 160 °C.