Influence of Minor Sc Addition on Microstructure and Mechanical Properties of Extruded Al-7Zn-2Mg-1.5Cu-0.1Zr Alloy in T6 Heat Treatment

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The effects of minor Sc on microstructure and mechanical properties of Al-7Zn-2Mg-1.5Cu-0.1Zr alloy under different aging time were investigated by means of tensile test, scanning electron microscopy and transmission electron microscopy, respectively. The results show that the aging strengthening tendency of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys exhibits the similar feature. After T6 treatment, the maximum values of ultimate tensile strength and yield strength are 700 MPa and 602 MPa, respectively. The tensile fracture of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys exhibits a mixed ductile-brittle fracture. The results of TEM observation show that large amounts of GP zones and η' phase distribute homogeneously inside the grains, and some rod-like precipitates distribute along grain boundaries in two alloys. Besides, there also exist lots of $\text{Al}_3(\text{Sc}, \text{Zr})$ phase in Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy with different treated states. The grain refinement strengthening and precipitation strengthening play the dominant role in the strength increase of the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy.
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1. Introduction

As a typical age-hardenable alloy, Al-Zn-Mg-Cu series alloys processed in the form of plate, extrusion or forging, are widely used for the aircraft and automobile parts in virtue of their properties of light mass, high specific strength and good ductility.^{1,2)} With the rapid development of modern industry, the demand for the performances of aluminum alloys as the structural materials is putting forward higher. The combined use of Zr and Sc elements is paid more attention to improve the performances of aluminum alloys.³⁻⁵⁾ Xu *et al.*⁶⁾ investigated the microstructure and properties of Al-Si alloy with Sc and Zr, and found that the addition of Sc can obviously refine the grains and reduce the secondary dendrite arm spacing. Ikeda *et al.*⁷⁾ investigated the effect of thermally stabilized particles on the recrystallization behavior of an Al-Mg-Si Alloy, and found that $\text{Al}_3(\text{Sc}, \text{Zr})$ particles may interfere with the recrystallization. Liu *et al.*⁸⁾ investigated the microstructure and properties of Al-Zn-Mg-Cu alloys with Sc and Zr, and found that $\text{Al}_3(\text{Sc}, \text{Zr})$ particles can effectively pin dislocations and sub-grain boundaries, and inhibit the occurrence of recrystallization.

Except for adjusting the content of main alloy elements and performing the microalloying treatment, the heat treatment is also a way to improve the performances of Al-Zn-Mg-Cu series alloys. Conventional T6 treatment includes solid solution treatment and aging treatment. During the aging treatment, precipitation of solute atoms takes place and acts as the key strengthening mechanism. It is generally believed that the precipitation sequence of this series aluminum alloys is as follows:⁹⁻¹¹⁾ Supersaturated solid solution $\rightarrow$ GP zones $\rightarrow$ metastable η' phase $\rightarrow$ stable η phase. Therefore, the reasonable aging time is imperative to regulate the species of precipitate and improve the size and distribution of precipitated phase, and thus enhance the mechanical properties of Al-Zn-Mg-Cu series alloys.

In this paper, the microstructure and mechanical properties of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys under different aging time were investigated. The aim of the present study was twofold. The first aim was to study the relationship between the microstructure characteristics and mechanical properties of Al-7Zn-2Mg-1.5Cu-0.1Zr alloy with Sc element addition. The second aim was to research the effect of aging time on the aging strengthening of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys. The strengthening mechanism and relationship between the microstructure and mechanical properties after T6 treatment were also discussed to provide reliable theoretical basis for the engineering application of this series of aluminum alloys.

2. Experimental Procedure

Two kinds of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) (mass%) alloys were applied for comparative research in this investigation. The alloy ingots were extruded into bars after homogenization treatment at 470°C for 12 h, where the extrusion temperature was 450°C and the extrusion ratio was about 40. The alloy bars were subjected to solution treatment at 475°C for 1 h, followed by water quenching, and then aged at 120°C for various time from 20 h to 56 h.

The room temperature tensile properties of two alloys under different aging time were measured to obtain the relationship between the aging time with tensile strength of alloys. The tensile tests were conducted on WDW 200E electron universal testing machine. The fracture surfaces of tensile specimens were characterized with S-3400N scanning electron microscope. In addition, the TEM observation observed on a JEM-2100 transmission electron microscope (TEM) with an acceleration voltage of 200 kV, and the samples for TEM observation were prepared with twin-jet electropolishing method at 21 V in a solution of 30% nitric acid and 70% methanol solution cooled to -30°C. Figure 1 shows the flow of the sample production process and experiment.

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Fig. 1 Production and experimental process of two alloys.

3. Results and Discussions

3.1 Mechanical properties

Figure 2 shows the changing curves of mechanical properties for two alloys with aging time. It can be noted from Fig. 2(a) that the ultimate tensile strength of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys increases with increasing aging time in the early stage of age-hardening, and the first strength peak is reached at 28 h and 32 h for the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc and Al-7Zn-2Mg-1.5Cu-0.1Zr alloys, respectively. With further prolonging the aging time, the strength of two alloys firstly decreases and then increases until the second strength peak is attained at 44 h and 48 h, respectively. It is obvious that the second strength peak of two alloys is the highest, and the corresponding strength values are 700 MPa and 505 MPa for the Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys. In addition, it can be seen from Fig. 2(b) that with increasing the aging time, the yield strength of two alloys also exists double peaks, and the yield strength of two alloys at second peak is significantly higher than that at first peak. The highest values of yield strength for the Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys are 602 MPa and 421 MPa. And the results indicate that the addition of Sc significantly promotes the appearance of the strength of alloy and the variation in the yield strength is in good accordance with variation tendency in the ultimate tensile strength for two alloys. This reason will be discussed in detail later. As shown in Fig. 2(c), the elongation to failure for two alloys decreases with increasing aging time, and the alloy without Sc has the higher elongation than Sc-containing alloy. The results of mechanical properties test indicate that two alloys all exhibit similar aging strengthening tendency. In whole aging period, two alloys display the double peak phenomenon.

3.2 Microstructural observation

Figure 3 shows the microstructures of two alloys subjected to T6 treatment. It can be noted that two alloys exhibit the difference in the grain size. And the different color indicates different grain orientation. The microstructure consists of equiaxed grains with an average size of 71.7 μm for Al-7Zn-2Mg-1.5Cu-0.1Zr alloy, and the sub-grain size in Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy is smaller with an average

Fig. 2 Effects of aging time on tensile properties of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloy: (a) Ultimate tensile strength; (b) Yield strength; (c) Elongation.

size of 2.8 μm . It is well known that the mechanical properties of polycrystalline metallic materials depend on the average grain size. The relationship between the mechanical properties and average grain size can be expressed by the Hall-Petch equation as follows,¹²⁾ eq. (1).

$$\sigma_s = \sigma_0 + kd^{-1/2} \quad (1)$$

where σ_0 is the intrinsic resistance of the lattice to dislocation motion and d is the average grain size, k is a material-dependent constant that represents the contribution of grain boundary to the related strengthening. It can be known from the Hall-Petch relationship that the tensile strength is closely

Fig. 3 The EBSD images of two alloys with solution plus aging treatment: (a) Al-7Zn-2Mg-1.5Cu-0.1Zr alloy; (b) Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy.

related to the grain size. The smaller grain size is, the higher the tensile strength will be. Compared with the Al-7Zn-2Mg-1.5Cu-0.1Zr alloy, the much finer grains can be attained in the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy because the formation of $\text{Al}_3(\text{Sc}, \text{Zr})$ phase can effectively block the movement of grain boundary and inhibit recrystallization.¹³⁾ Therefore, the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy has the higher tensile strength.

The TEM images of precipitates in two alloys with T6 states are shown in Fig. 4 and Fig. 5. It can be seen that a large number of fine precipitates form homogeneously inside the grains for two alloys under different aging time. These fine precipitates were identified as nano-scaled GP zones and η' phase in some reported results.¹⁴⁾ Furthermore, some $\text{Al}_3(\text{Sc}, \text{Zr})$ phase with the bean-like shape can be seen inside the α -Al matrix due to the addition of Sc element, and the $\text{Al}_3(\text{Sc}, \text{Zr})$ phase also exhibits a larger average size than the GP zones and η' phase, as shown in Fig. 5. And the SAED patterns for the Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloy aged for 28 h are shown in Fig. 4(d) and Fig. 5(d). The diffraction spots at $\{1, 3/4, 0\}$ positions are corresponding to the diffraction feature of GP zones. With prolonging the aging time, except for the diffraction spots of GP zones, other ones reflecting the η' phase are detected at $2/3$ of $\{220\}$ positions shown in Fig. 4(e) and Fig. 5(e). It indicates that the η' precipitate has formed after aging for 44 h. After the aging time reached 56 h, the diffraction spots of GP zones disappear shown in Fig. 4(f) and Fig. 5(f), indicating that the GP zones are all transformed into η' phase.

Fig. 4 TEM images of precipitates in Al-7Zn-2Mg-1.5Cu-0.1Zr alloy under different aging time: (a, d) 28 h; (b, e) 44 h; (c, f) 56 h.

Fig. 5 TEM images of precipitates in Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy under different aging time: (a, d) 28 h; (b, e) 44 h; (c, f) 56 h.

In addition, the TEM images in Fig. 6 show the grain boundaries of two alloys under different aging time. The precipitated phase continuously distributes at the grain boundaries for two alloys aged for 28 h. Meanwhile, the precipitate free zone (PFZ) appears at the boundary and its width is about 26.7 nm and 30.8 nm for two alloys, respectively. With prolonging the aging time, the precipitate at the grain boundary grows up, the short rod-like particles gradually form along the grain boundaries and the width of PFZ increases to 32.1 nm and 37.6 nm for two alloys aged for 44 h. The phase further coarsens and distributes discontinuously at the grain boundaries and the width of PFZ further increases to 42.3 nm and 49.8 nm for two alloys aged for 56 h.

It can be found through the comparison that there is almost no difference at the size and density of GP zones and η' phase inside the grains between the Al-7Zn-2Mg-1.5Cu-0.1Zr alloy and Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy, but the size of precipitate at the grain boundaries is smaller and the width of PFZ is wider in the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy. Therefore, the aging strengthening tendency of two alloys is similar, and the alloy with the wider PFZ exhibits the lower elongation.

Figure 7 shows the $\text{Al}_3(\text{Sc, Zr})$ phase in the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy with different treated states. The $\text{Al}_3(\text{Sc, Zr})$ phase still exists after solution treatment, and distributes near the sub-grain boundaries, as shown in

Fig. 7(a). It indicates that the $\text{Al}_3(\text{Sc, Zr})$ phase exhibits an excellent thermal stability and plays an important role in pinning the grain boundaries, which contributes significantly to inhibit the growth of grains in Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy. Besides, there also exists smaller $\text{Al}_3(\text{Sc, Zr})$ particle inside the grains, as shown in Fig. 7(b). It can be thought that these $\text{Al}_3(\text{Sc, Zr})$ particle with the smaller size should precipitate during the aging treatment. It can be suggested that the enhancement in the strength of Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy can also relate to the formation of $\text{Al}_3(\text{Sc, Zr})$ phase. The reinforcing effect of $\text{Al}_3(\text{Sc, Zr})$ phase in the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy mainly concerns the dispersal-strengthening and fine-grain strengthening which has been mentioned above.

The dispersion-strengthening effect includes the shearing mechanism for the precipitates with smaller size and Orowan bypass mechanism for the precipitates with larger size, respectively. Therefore, the $\text{Al}_3(\text{Sc, Zr})$ phase with the larger size than η' phase provides the reinforcement mainly by the Orowan bypass mechanism. The augment in the yield strength ($\Delta\sigma_{or}$) related to the Orowan mechanism can be expressed as follows,¹⁵⁾ from eq. (2) to eq. (4).

$$\Delta\sigma_{or} = \frac{k_4 M G b}{\lambda \sqrt{1-\nu}} \ln\left(\frac{d_s}{b}\right) \quad (2)$$

$$d_s = \frac{\pi d_m}{4} \quad (3)$$

Fig. 6 TEM images of grain boundaries for two alloys under different aging time: Al-7Zn-2Mg-1.5Cu-0.1Zr alloy (a) 28 h, (b) 44 h, (c) 56 h; Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy (d) 28 h, (e) 44 h, (f) 56.

$$\lambda = \left[\frac{1}{2} \sqrt{\frac{2\pi}{3f_v}} - 1 \right] \frac{\pi d_m}{4} \quad (4)$$

where M is the Taylor factor which generally depends on the texture and the orientation of tensile axis, ν and G is the Poisson's ratio and the shear modulus of the Al-matrix, b is the Burgers vector of mobile dislocations in the Al matrix, k_4 is a constant which depends on the size and distribution of second phase particles, f_v is the volume fraction of the particles, and d_s and λ are the mean particle diameter and an effective inter-particle spacing on the slip plane of dislocations, respectively. This theory suggests that the smaller the size of the particle and the larger the volume fraction of the particle, the higher the tensile strength and hardness will be. Therefore, the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy with different sizes of $\text{Al}_3(\text{Sc}, \text{Zr})$ phase exhibits the much higher tensile strength than the Al-7Zn-2Mg-1.5Cu-0.1Zr alloy.

3.3 Fracture characteristics observation

The morphologies of tensile fracture surfaces for two alloys with the second peak aging stage are shown in Fig. 8. Obviously, the numerous dimples can be observed on the fracture surfaces of two alloys. In addition, the cleavage characteristic can be also noted on the fracture surface. It implies that the tensile fracture mode of two alloys is the mixed ductile-brittle fracture. And it is found through

comparing Fig. 8(a) with Fig. 8(b) that the dimples for the Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy are smaller and shallower than those for the Al-7Zn-2Mg-1.5Cu-0.1Zr alloy. For these materials with low strength and good plasticity, the necking usually occurs during uniaxial tension deformation. The dimples tend to grow in the necking areas where the concentrated stress is large, so that the size of dimple on the fracture surface is larger and deeper. In contrast, for those materials with high strength and low plasticity, the necking phenomenon is difficult to be noted during uniaxial tension deformation. Because the necking is not obvious, the stress concentration is small and the dimples have a similar growth rate over a larger range. Correspondingly, the dimples on the fracture surface are smaller and shallower.¹⁶⁻¹⁸⁾ This corresponds to the lower elongation of Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy.

4. Conclusion

- (1) The aging strengthening tendency of Al-7Zn-2Mg-1.5Cu-0.1Zr and Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloys subjected to T6 treatment is similar.
- (2) The tensile fracture mode of Al-7Zn-2Mg-1.5Cu-0.1Zr(-0.2Sc) alloys subjected to T6 treatment is a mixed ductile-brittle fracture.
- (3) The increment in the tensile strength of Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy aged for different time mainly

Fig. 7 $\text{Al}_3(\text{Sc}, \text{Zr})$ phase in Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy with different treated states: (a) solid-solution treated state; (b) aging treated state.

Fig. 8 Morphologies of tensile fracture surfaces for two alloys with the second peak aging stage: (a) Al-7Zn-2Mg-1.5Cu-0.1Zr alloy; (b) Al-7Zn-2Mg-1.5Cu-0.1Zr-0.2Sc alloy.

derives from the dispersal-strengthening and fine-grain strengthening caused by the $\text{Al}_3(\text{Sc}, \text{Zr})$ phase.

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