

Effects of Er and Zr additions on precipitation and recrystallization of Al-Mg alloy

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Abstract. The effects of Er and Zr on the precipitation and recrystallization of Al-Mg alloy was investigated using hardness testing, optical microscopy and transmission electron microscopy. The results show that Al-Mg-Er-Zr alloy has a markedly hardening effect during isochronal annealing. The recrystallization temperature of the Al-Mg-Er-Zr alloy is significantly higher than that of Al-Mg alloy, owing to the presence of nanosized and coherent Al₃(Er,Zr) precipitates.

Introduction

Aluminium alloys with magnesium as the major alloying element constitute a group of non-heat treatable alloys with medium strength, high ductility, excellent corrosion resistance and weldability. Unlike the heat treatable alloys, these materials derive their strength primarily from solid solution strengthening by Mg and strain hardening. However, high levels of Mg cause processing challenges and the susceptibility to stress corrosion cracking [1,2]. An effective alternative method, minor-alloying, addition of small amount of alloying elements such as Sc and Zr, is used to increase the strength and thermal stability of Al-Mg alloys by the formation of Al₃Sc and Al₃(Sc,Zr) dispersoids [3-5].

The heavy rare-earth element Er, which is similar to Sc but more cheaper, has been shown to improve the mechanical properties by the formation of Al₃Er [6-8]. A recent study reports that there is a synergetic effect of Zr and Er on the precipitation hardening of Al-Er-Zr alloy [9]. Li and coworkers [10] also found that the recrystallization temperature of the Al-Er-Zr alloy is significantly higher than that of Al-Er alloy, owing to the presence of nanosized and coherent Al₃(Er,Zr) precipitates. In this paper, we focus on quaternary Al-Mg-Er-Zr alloy and investigate the precipitates strength and the subsequent effect on recrystallization resistance after cold rolling.

Experimental materials and methods

The experimental alloys were prepared through a conventional melting (780°C) and casting (720°C) route. The composition of the alloys, analysed by X-Ray Fluorescence (XRF), is given in Table 1. The as-cast alloys were annealed in air isochronally from 150°C, terminating at 550°C, with increments of 25°C, each lasting 3h. After isochronally annealing, the peak annealed samples were cold-rolled to 80% reduction. Then the as-rolled samples were annealed in air at temperatures between 125°C and 525°C, at increments of 50°C, and at each temperature point the samples were isothermally aged for 1 h.

The Vickers microhardness was measured on polished samples with a load of 200 g and a dwell time of 10 s. Samples polished using standard metallographic techniques and etched with Keller reagent were used for optical microscopy (OM) characterization. Thin foils for TEM observation were prepared by twin-jet polishing with an electrolyte solution consisting of 25% HNO₃ and 75% methanol at -30°C. The specimens were analyzed using a JEOL 2100 microscope with an operating voltage of 200 kV.

Table 1. Chemical composition of the alloys (wt.%).

| Material | Mg | Er | Zr | Al |
|---------------------|-----|------|------|---------|
| Al-5Mg | 5.1 | 0 | 0 | Balance |
| Al-5Mg-0.3Er-0.25Zr | 4.8 | 0.29 | 0.25 | Balance |

Results and Discussion

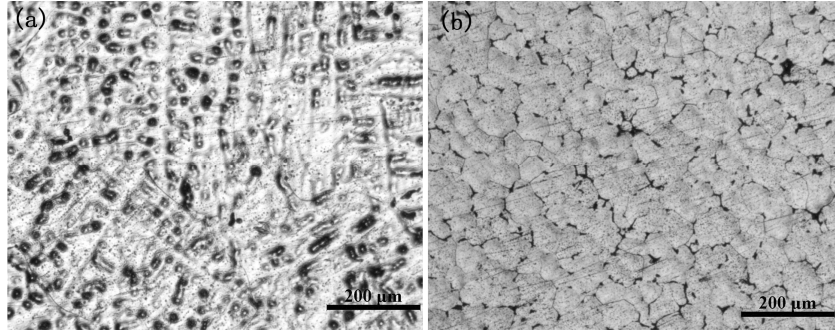


Fig. 1. As-cast microstructures of the alloys: (a) Optical microstructures of the Al-Mg alloy; (b) Optical microstructures of the Al-Mg-Er-Zr alloy

Microstructure of as-cast alloys. The microstructures of the as-cast Al-Mg alloys are shown in Fig. 1. It can be seen that an dendritic structure, which is the result of segregation of solute at the interdendritic regions, in the Al-Mg alloy. But the grains were refined and dendrite structures disappeared by the addition of Er and Zr simultaneously. Previous research also had shown that with addition of Er alone can refine the dendrite structures but can not eliminate it completely. This indicates that by joint addition of Zr and Er have a stronger refining effect than addition of Er singly.

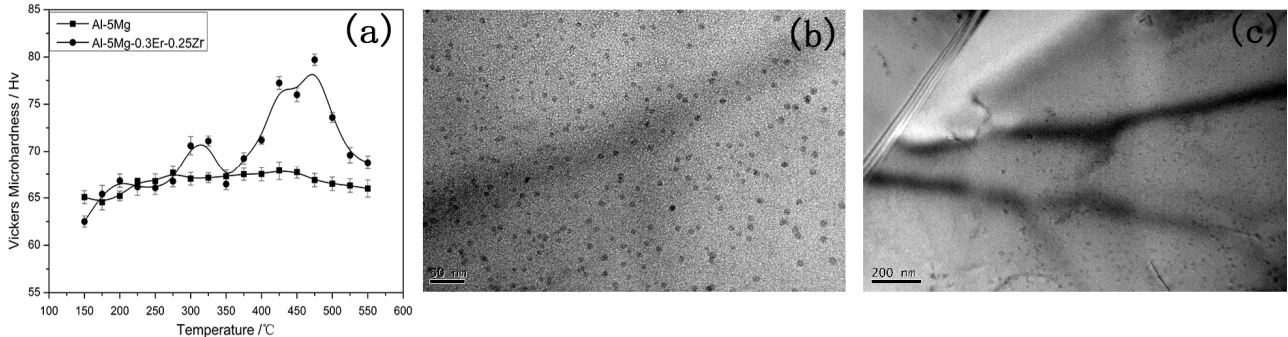


Fig. 2. The hardness curve of isochronal annealing(a) and the TEM micrograph of Al-Mg-Er-Zr alloy at the hardness peak value(b),(c).

Precipitation hardening. Fig. 2.(a) displays the hardness evolution of Al-Mg and Al-Mg-Er-Zr alloys during isochronal annealing. The Al-Mg alloy has no annealing hardening effect, its hardness remaining almost constant for all temperatures. The hardness of Al-Mg-Er-Zr alloy has a very significant annealing strengthening effect due to produce the second phase, $Al_3(Er,Zr)$, in the matrix. The hardness of Al-Mg-Er-Zr reaches the first peak of about 70 Hv between 300 and 325°C, then has a slightly decreases between 325 and 350°C. The hardness of Al-Mg-Er-Zr reaches the senond peak of about 80 Hv between 425 and 475°C. Due to the relative high diffusivity of Er in α -Al, so Al_3Er firstly precipitate from α -Al matrix between 300 and 325°C. On the other hand due to a synergetic effect of Zr and Er on the precipitation hardening, the remaining Er can stimulate the decomposition of Zr and lead to a significant anneal hardening between 425 and 475°C.

The TEM observations of Al-Mg-Er-Zr alloy after isochronal annealing to 475°C are shown in Fig. 2.(b),(c) Fine dispersed precipitates were observed in the Al-Mg-Er-Zr alloy. According to former study these precipitates should be $Al_3(Er,Zr)$. The precipitates are homogeneously distributed in the intergrain area, but at the grain boundary there exist a precipitates free zone which indicates that due to form some primary phase at the grain boundary thereby the content of solute atoms in the matrix decrease. The average radius of these small precipitates is about 10 nm. In this

size these small precipitates should be coherent with the matrix. So these precipitates can lead to the very significant annealing strengthening effect.

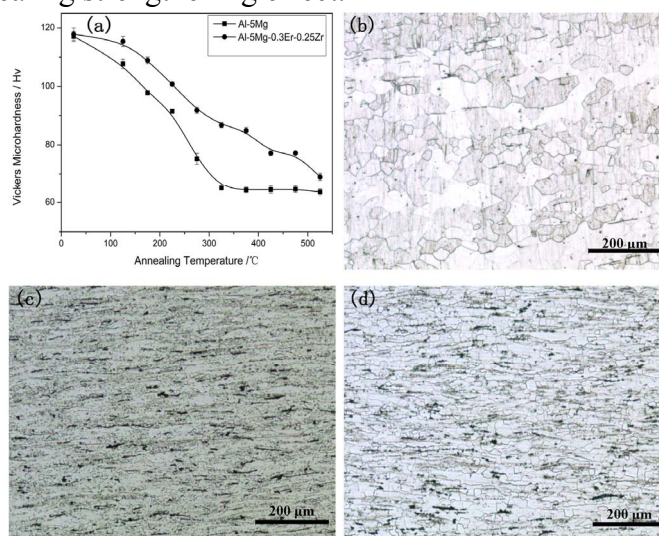


Fig. 3. (a) Hardness as a function of annealing temperature for Al-Mg and Al-Mg-Er-Zr alloys and micrographs of the cold-rolled alloys; (b) Al-Mg alloy annealed at 325°C for 1h; (c) Al-Mg-Er-Zr alloy annealed at 375°C for 1h; (d) Al-Mg-Er-Zr alloy annealed at 525°C for 1h

Recrystallization Resistance. For investigating the recrystallization resistance of alloys, the cold-rolled samples were annealed in the temperature range of 125–525 °C, at increments of 50 °C, and at each temperature point the samples were isothermally annealed for 1 h. Fig. 3.(a) shows the average microhardness of cold-rolled Al-Mg and Al-Mg-Er-Zr alloys as a function of temperature. It may be seen that the hardness of Al-Mg-Er-Zr alloy declines slowly with increasing the annealing temperature while that of Al-Mg alloy declines obviously from 225°C to 325°C. The annealed microstructures of cold-rolled Al-Mg and Al-Mg-Er-Zr alloys are compared in Fig. 3.(b),(c),(d). It can be seen that Al-Mg alloy is fully recrystallized after annealing at 325°C for 1 h, whereas Al-Mg-Er-Zr alloy basically keeps a fibrous structure after annealing at 375°C for 1 h, which corresponds to the difference in hardness between Al-Mg and Al-Mg-Er-Zr alloys. The microhardness of Al-Mg alloy decreases to 65Hv at 325°C, while Al-Mg-Er-Zr maintains a relatively high microhardness of 85.1Hv at 375°C. Al-Mg-Er-Zr alloy displays a partially unrecrystallized structure even at 525°C. In present Al-Mg-Er-Zr alloy, due to the addition of Er and Zr can form $\text{Al}_3(\text{Er,Zr})$ precipitates during isochronal annealing. The $\text{Al}_3(\text{Er,Zr})$ precipitates with large number density and small radius, should be favored to attain a large Zener drag which retards the movement of dislocation and subgrain boundaries. Thus the Al-Mg-Er-Zr alloy possess remarkable recrystallization resistance as compared with Al-Mg alloy, which shows great potential for the development of thermally stable aluminum alloys.

Summary

The combined addition of Er and Zr to Al-Mg alloy results in dramatically refining effect, significant precipitation strengthening effect and remarkable recrystallization resistance. The recrystallization temperature of the Al-Mg-Er-Zr alloy is significantly higher than that of Al-Mg alloy, this can be attributed to the tiny and coherent $\text{Al}_3(\text{Er,Zr})$ precipitates, which precipitation during isochronal annealing.

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