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Effects of Er on Microstructures and Properties of Al-Zn-Mg Alloys

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Abstract

The ternary Al-Zn-Mg system has been extensively investigated due to its technological importance as a basis for the high-strength 7000 series aluminium alloys. In this paper, six different compositions of Al-Zn-Mg alloys containing rare earth element Er were prepared by ingot metallurgy. The effects of Er on the microstructures and properties of Al-Zn-Mg alloys were studied by hardness test, tensile properties measurement, X-ray diffraction analysis (XRD), optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy dispersive x-ray spectroscopy(EDXS) analysis.

1. Introduction

The role of rare-earth (RE) in aluminium based alloys is well studied and can be summarized as follows: 1) grains refinement, 2) segregation minimization, 3) pores and impurities remover, 4) mechanical properties improvement [1-2]. Many studies have shown that the RE element Sc is by far the most effective RE element for property improvement of aluminum alloys, but the main drawback of Sc is that it is very expensive. In our earlier studies [3-8], considerable efforts have been made to identify a substitute of Sc. Trace element Er was added to high-pure Al, Al-3Mg and Al-5Mg (wt%) alloys. The experimental results show that for all the alloys studied, with the addition of Er, grains can be remarkably refined, mechanical properties can be improved, and the recrystallization can be inhibited apparently. Adding Er to the Al-3Mg alloy, the eutectic compound is refined and disperses more homogeneously, which improves the quality of the castings. In a word, the element Er has a similar effect with Sc on Al and Al alloys and its price is just only 1/40 that of Sc. With further investigation about the effect of Er on aluminum alloys, we are likely to be able to exploit new advanced aluminum alloys containing Er.

In the present investigation, we continue our work concerning the effects of Er on microstructure and properties of the Al-Zn-Mg alloys, which is an important group of age-hardenable alloys and is widely used for structural applications in aerospace and automotive industry due to their high specific mechanical properties [9].

2. Experimental Procedures

The compositions of materials used in the investigation are provided in Table 1. These alloys containing Er were prepared by a casting metallurgy method, with high pure Al

(99.99%), commercial pure Mg(99.9%), commercial pure Zn(99.9%) and Al-6.2Er master alloy. All the castings were homogeneously annealed at 470°C for 13 hours and then hot-rolled and cold-rolled into 2mm thick sheets. The samples for hardness test were annealed in an air-circulating furnace in the temperature range of 200-400℃ (got an point every other 25℃), keeping for an hour, then cooling down in air. The tensile specimens were solution heat treated at 475℃ for 30min in an salt bath furnace followed by quenching in room temperature water and then aged at 120℃ for 24h.

The tensile properties were tested in an INSTRON4302 Material Testing Machine; the hardness of each sample was measured in an HBWUV-187.5 hardness tester. Samples for optical microscope observation were electrolytically polished and anodized in Baker's reagent. The observation was carried out with a NEOPHOT-21 optical microscope made in Germany. Samples for TEM examination were prepared by a twin-jet electropolishing method in a solution of 25% nitric acid and 75% (vol%) methanol, and examination was performed in a H-800 TEM. The distribution of Er in the Al-Zn-Mg alloys was investigated using X-ray diffraction (XRD) and a HITACHI S-3500N scanning electron microscope (SEM).

Table 1: Nominal component of the studied Al-Zn-Mg alloys (wt.%).

Alloy number	Er	Zn	Mg	Al
1#	0	6.0	2.0	Bal.
2#	0.10	6.0	2.0	Bal.
3#	0.25	6.0	2.0	Bal.
4#	0.40	6.0	2.0	Bal.
5#	0.55	6.0	2.0	Bal.
6#	0.70	6.0	2.0	Bal.

3. Results and Discussions

3.1 Effect of Er on Mechanical Properties

The effect of Er content on tensile properties of the Al-Zn-Mg alloys at room temperature, under cold rolled and aged state (120℃/24h) respectively, is shown in Figure 1. It can be seen that the tensile strength σ_b and the yield strength $\sigma_{0.2}$ of the alloy increase remarkably with Er addition. For example, the σ_b and the $\sigma_{0.2}$ of the Al-Zn-Mg-0.7%Er alloy aged at 120 ℃/24h improved about 100MPa compared to that of the Al-Zn-Mg alloy free of Er. When the Er content is 0.1 wt%, the increment in strength is the largest, but the increment reduces with an increase in the Er content, though the strength continues to increase compared with the Al-Zn-Mg alloy free of Er. It is notable that the elongation of the alloys decreases a little with Er additions.

3.2 Effect of Er on As-Cast Microstructure

The addition of Er to Al-Zn-Mg alloy has a significant effect on the as-cast microstructure. Figure 2 (a) shows that grains of the Al-Zn-Mg alloy without Er addition are rather coarse and there is apparent dentrite structure. When adding 0.1%Er to the Al-Zn-Mg alloy, the grains are remarkably refined and most of the dentrite structure disappears. As for Al-Zn-Mg-0.4Er and Al-Zn-Mg-0.7Er, grains become rather small and the dentrite structure almost disappears.

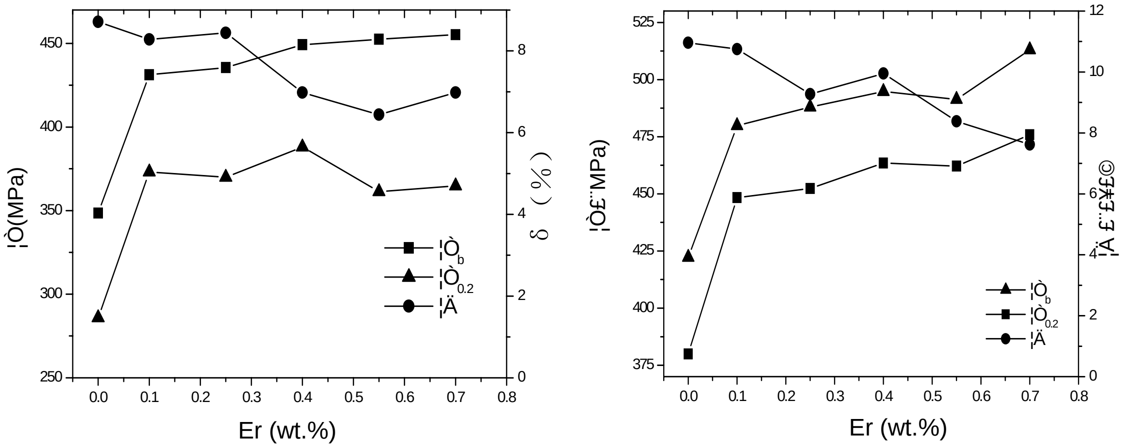

Figure 1: Relationship between mechanical properties and Er content. a) Cold rolled, and b) aged at 120℃ for 24h.

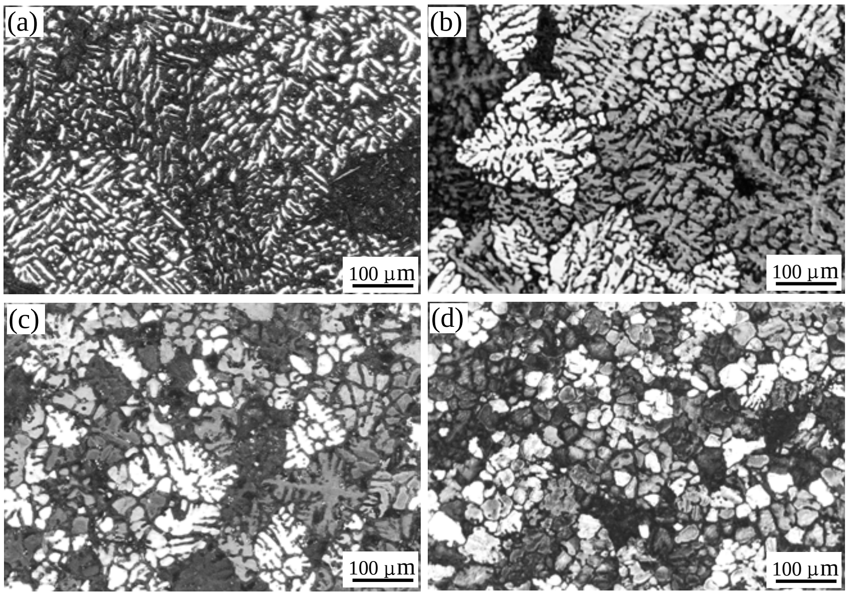

Figure 2: Microstructure of as-cast Al-Zn-Mg-(Er) alloys showing that Er additions have a strong grain refinement effect. (a) Al-Zn-Mg, (b) Al-Zn-Mg-0.1Er, (c) Al-Zn-Mg-0.4Er, and (d) Al-Zn-Mg-0.7Er.

In order to investigate the existent form of Er in Al-Zn-Mg alloys, XRD analysis was carried out and patterns obtained from Al-Zn-Mg and Al-Zn-Mg-0.4Er alloys are shown in Figure 3, in which A was from Al-Zn-Mg alloy, and B from the Al-Zn-Mg-0.4Er alloy.

The major phases in the Al-Zn-Mg alloy are α-Al, $MgZn_2$ and $Mg_{32}(Al,Zn)_{49}$, Figure 3A. But there is an additional phase Al_3Er in the Al-Zn-Mg-0.4Er alloy besides phases α-Al, $MgZn_2$ and $Mg_{32}(Al,Zn)_{49}$, Figure 3B. This indicates that Er exists in the form of Al_3Er in the Al-Zn-Mg alloy. Figure 4 shows the SEM observation and the point-located energy analysis of the as-cast Al-Zn-Mg-0.4Er alloy, which confirmed the existence of Al_3Er in Al-Zn-Mg alloy.

The refining of the as-cast microstructure of the Al-Zn-Mg alloy is likely due to the primary Al_3Er particles formed during solidification. When adding trace Er to the Al-Zn-Mg alloys, Er seems to react only with Al and form the Al_3Er phase, whose crystal structure ($L1_2$) is very close to that of the Al matrix, and the mismatch is rather small (about 4.1%). Therefore, Al_3Er particles are coherent or semi-coherent with the Al matrix, which makes the fine primary Al_3Er particles (as shown in Figure 4) act as heterogeneous nucleation sites, resulting in remarkable grain refining.

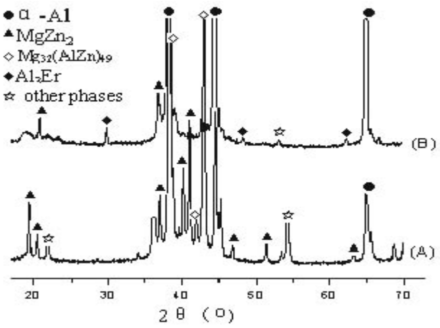

Figure 3: XRD patterns of (A) Al-Zn-Mg, and (B) Al-Zn-Mg-Er alloys.

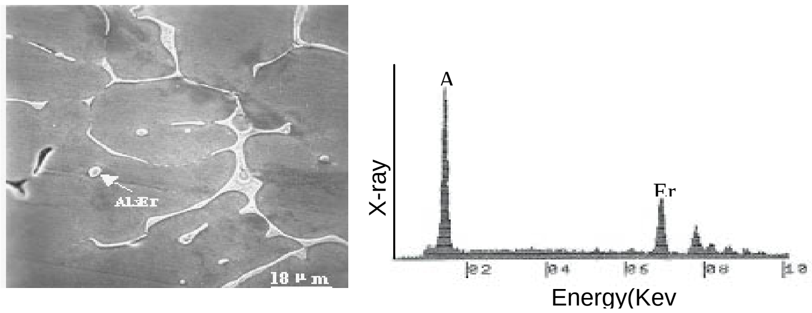

Figure 4: SEM micrograph showing as-cast Al-Zn-Mg-0.4Er alloy and EDXS obtained from the arrowed point in the micrograph.

3.3 Effect of Er on Recrystallization Behavior

In order to study the effect of Er addition on the crystallization behavior of the Al-Zn-Mg alloy, the cold rolled sheets were annealed in a range of 250~450℃ and performed hardness tests. The relationship between hardness and annealing temperature is shown in Figure 5, T_s and T_f corresponding to the starting temperature and ending temperature of crystallization, respectively.

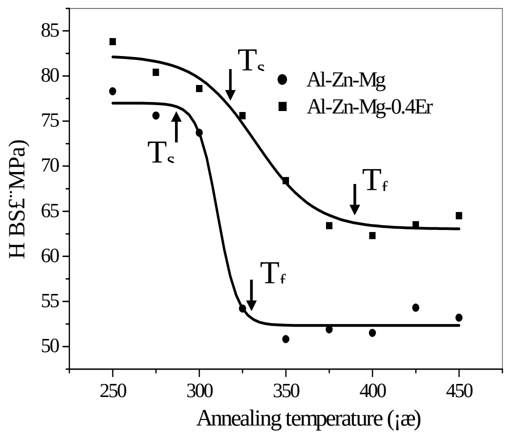

Figure 5: Relationship between hardness and annealing temperature. It also shows the effect of Er on crystallization behavior of Al-Zn-Mg alloys.

From Figure 5, it can be seen that the addition of 0.4%Er not only improves the hardness of the Al-Zn-Mg alloy, but also increases the recrystallization starting temperature and the ending temperature about 50℃, which suggests that Er can prevent the recrystallization of Al-Zn-Mg alloy, and thus improve the thermal stability of the alloy. This can be confirmed by the following microstructure observation.

The microstructure of the Al-Zn-Mg alloy and the Al-Zn-Mg-0.4Er alloy, annealed at 325℃ and 425℃ for an hour, (see Figure 6 (a)-(d)) revealed that recrystallization has begun clearly in the Al-Zn-Mg alloy after annealed at 325℃ for an hour(Figure 6 (a)), but for Al-Zn-Mg-0.4Er alloy, it didn't appear (Figure 6 (b)). When annealed at 425℃ for an hour, the recrystallized structure of the Al-Zn-Mg alloy is apparently coarsened (Figure 6(c)). Although full recrystallization has also happened in the Al-Zn-Mg-0.4Er alloy, fine equiaxial grains still exist in the alloy (Figure 6 (d)), which suggests that Er addition can refine the recrystallized structure.

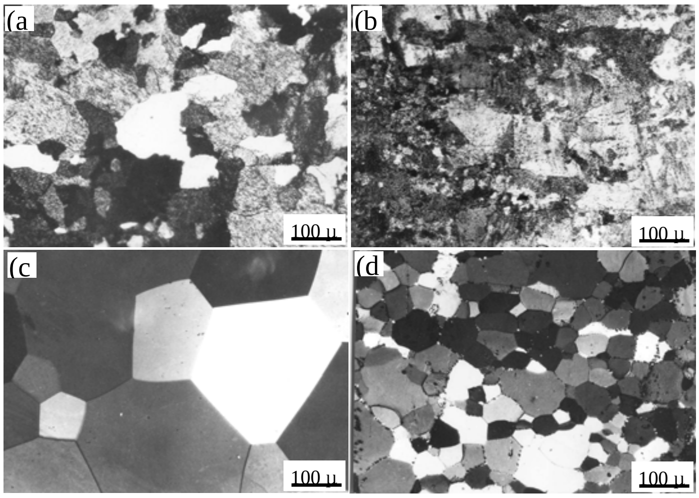

Figure 6: Recrystallization microstructure of Al-Zn-Mg alloys annealed for 1h at different temperatures. (a) Al-Zn-Mg alloy, 325℃ (b)Al-Zn-Mg alloy, 425℃ (c)Al-Zn-Mg-0.4Er alloy, 325℃ (d)Al-Zn-Mg-0.4Er alloy, 425℃

The improvement of the recrystallization temperature of the Al-Zn-Mg alloy is probably due to the second-born Al_3Er particles(as shown in Figure 7,arrowed), which precipitated from the matrix. These fine dispersed Al_3Er particles have a rather strong function of pinning the dislocations and sub-boundaries, preventing the movement of dislocations and the incorporation of sub-boundaries, thus inhibited the recrystallization of the Al-Zn-Mg alloy.

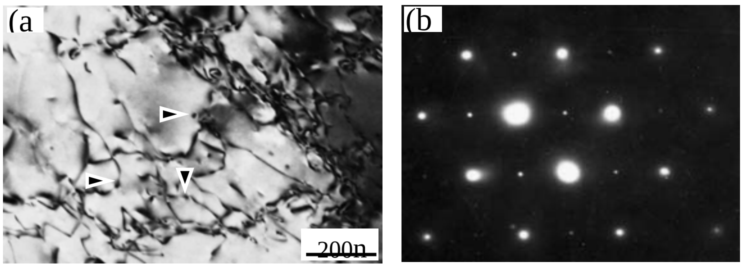

Figure 7: (a) TEM image of the Al-Zn-Mg-0.4Er alloy annealed for 1h at 350℃, showing that dislocations pinned by fine Al_3Er particles (b) electron diffraction pattern taken from matrix regions containing such fine precipitates (arrowed in (a))

4. Conclusions

1) The tensile strength σ_b and the yield strength $\sigma_{0.2}$ of Al-Zn-Mg alloys increase significantly, and there is little decrease in elongations, with Er additions.
2) The recrystallization temperature of the Al-Zn-Mg alloy can be increased by about 50℃ by adding trace amounts of rare earth element Er.
3) The addition of Er to the Al-Zn-Mg alloy has a significant effect on the as-cast microstructure, i.e. grains can be remarkably refined and the dentrite structure almost disappears. This probably can be interpreted that the fine primary Al_3Er particles formed during solidification act as the nucleation sites.

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