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Rolling route for refining grains of super light Mg-Li alloys containing Sc and Be

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Abstract: Pioneering work on Sc or/and Be added Mg-Li alloys with refined grains was initiated. Various rolling-based thermo-mechanical treatments on these Mg-Li alloys were carried out. Four Mg-Li alloys were prepared by vacuum melting process. A unique route for producing fine grains was applied which concluded solution treatment at 350 °C, cold rolling with 60% thickness reduction and 250 °C annealing, successively.

Key words: Mg-Li alloy; beryllium; scandium; solid solution strengthening

1 Introduction

Magnesium alloys have the advantages of low density, high specific strength, heat conduction, damping and electro-magnetic wave shielding capacities, which makes Mg alloys very popular in 3C (computer, consumer) electronic communication and and transportation industries. However, it is commonly recognized that magnesium possesses poor formability because of its hexagonal close-packed structure. To make up this shortcoming and further reduce its weight, alloying magnesium with extremely low density element lithium (0.534 g/cm^3) can achieve the goals. For example, LAZ1110 alloy (Mg-11%Li-1%Al-0.5%Zn) possesses a low density of 1.64 g/cm³, as calculated by the rule of mixture[1], surprisingly, the actual density is 1.46 $g/cm^{3}[2]$, which is comparable to plastic materials. LEE et al[3] worked on various Mg-Li alloys and found that they were very ductile and their strengths were sufficient but not impressive. It is then conceived to modify these alloys with addition of Be and Sc to improve the strength and other fringe properties. Actually, a small addition of Be is common in preparing Mg alloys for de-oxygenation and preventing fire hazard during the molten casting process. However, the presence of Be in the Mg-Li alloys and the resulted effect were seldom studied. Sc is generally considered effective in refining grains and forming coherent precipitates for strengthened Al alloys[4]. It is then interesting to understand whether

it plays the same role in Mg alloys, specifically Mg-Li alloys. However, the knowledge in this respect is obscure and needs to be elucidated.

For superplastic metallic materials, fine-grained structure is a necessary quality in general. In present available literatures, however, presumably no typical fine grain structure in Mg-Li, especially in single phase Mg-Li were located. FURUI et al[5] reported a Mg-8%Li alloy with high superplastic elongation of about 1 780% and no typical fine grains were found after equal channel angular extrusion (ECAE). Similarly, DONG et al[6] worked on a Mg-8.5%Li alloy by high ratio extrusion to obtain medium superplasticity (about 527%), but no fine grain structure was found, which was in agreement with other reports [7-10] on the alloys with similar Li content. The above alloys with Li content between 8% and 9.5% have a dual phase structure $(\alpha + \beta)$. When the content of Li is higher than 11%, only a single phase (β) exists in Mg-Li alloys[11], which easily leads to grain growth, thus is unfavorable for the superplasticity. SIVAKESAVAM et al[12] reported a Mg-11.5Li-1.5Al alloy with coarse grains and an elongation of about 240%. LIU et al[13] provided in-depth TEM microstructure analysis of a ECAE processed Mg-14%Li alloy, however, no fine grains were observed. In this work, various thermo-mechanical processes based on cold rolling were carried out on four Mg-11Li-1Al-0.5Zn alloys for determining a route of producing fine-grained structure alloy.

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Four Mg-Li alloys were prepared in *d*200 mm cylindrical ingots by vacuum melting: basic alloy LAZ110 with a nominal composition of Mg-11%Li-1%Al-0.5% Zn; an alloy with only Sc addition to LAZ110; and an alloy with both Sc and Be addition to LAZ110. The four alloys were then homogenized at 350 °C, followed by an extrusion at 350 °C. The compositions were determined by Induction Coupled Plasma(ICP)- AES and Spark-OES instruments, as listed in Table 1. These plates were rolled to reduce the thickness by 30%, 60% and 90%, respectively, and each rolling pass adopted an approximate thickness reduction of 10%. Another deviated process route was: the plates were solution treated at 350 °C for 1 h and quenched in water

Table 1 Compositions of Mg-Li alloys studied (mass fraction, %)

Alloy	Li	Al	Zn	Sc	Be	Mg
LAZ1110	11.2	0.95	0.43	-	-	Bal.
LAZ1110+Sc	10.5	1.15	0.54	0.009	-	Bal.
LAZ1110+Be	10.4	1.02	0.66	_	0.017	Bal.
LAZ1110+Be+Sc	11.2	0.99	0.48	0.012	0.007	Bal.

before rolling. After cold rolling with the same reduction as adopted above, specimens were annealed at various temperatures from 50 to 250 °C. Specimens under various thermo-mechanical conditions were prepared for OM and SEM observation, X-ray diffraction analysis and mechanical properties tests.

3 Results and discussion

3.1 Microstructure after cold rolling

3.1.1 LAZ1110

The microstructure of extruded LAZ1110 predominantly exhibits coarse-grained β phase with bcc structure, as shown in Fig.1(a). Another fine grained α phase of small quantity is also present. Both α and β phases are identified in XRD analysis (Fig.2). Subsequent rollings with thickness reduction by 30%, 60% and 90% gradually and progressively elongate the grains, as shown in Figs.1(b)–(d).

3.1.2 LAZ1110+Sc

When a minute amount of Sc is added to LAZ1110, the grains are refined as comparing the microstructures in Figs.1 and 3. It is also noted that the amount of α phase increases with rolling reduction, which indicates that the β phase with bcc structure is not stable. With 11% (mass fraction) addition of Li, the mole ratio of Mg/Li is 2:1. Thus, Mg (hcp structured in most Mg



Fig.1 Optical micrographs of LAZ1110 alloy: (a) as-extruded state, showing β phase grain of about 51 µm and about 2.2% small distributive α particles; (b) 30%, (c) 60% and (d) 90% rolling reduction



Fig.2 XRD pattern of extruded specimen

alloys) is very capable of dissolving Li. However, the β phase with bcc structure is the original crystal of Li element. Thus, the β phase is not stable, external assistance such as room temperature aging, cold rolling or annealing, would expel Mg atoms to precipitate α phase[6].

3.1.3 LAZ1110+Be

This alloy contains 0.017% Be (mass fraction), which produces two micro-structural effects, grain coarsening and Widmanstatten structure (see Fig.4). The grain size in as-extruded state alloy is about 75 μ m, and there is a uniform spread of dots. Elongated grains are observed in the specimens with rolling reduction of 60%

and 90%, respectively.

3.1.4 LAZ1110+Sc+Be

This alloy contains 0.012% Sc and 0.007% Be (mass fraction). In as-extruded state alloy(Fig.5), the grain size is comparable with that of the previous LAZ1110+Sc. This result confirms the role of Sc in grain refining as it impedes the grain growth tendency in the presence of Be. None of the above alloys shows fine grain structure even if the samples are cold-rolled and subsequently annealed. However, an extra procedure of solid solution treatment dramatically alters the morphology, cold rolling alone does not offer sufficient strain energy to facilitate recrystallization, while solution-treatment gains some strain energy in this stage, then the proceeding of cold rolling immediately adds up more energy. By this procedure, the final stage of annealing leads to recrystallization with fine grains.

3.2 Strengthening and grain refining by solid solution treatment

After solution treatment followed by water quenching, the four alloys unanimously form a super-saturated β solid solution, as shown in Fig. 6. In XRD analysis, this single phase is confirmed, as shown in Fig.7. These solution-treated alloys show a significant increase of more than 200 MPa in strength compared to the as-extruded ones, as shown in Fig.8. This is explained that: the solution treatment strengthening



Fig.3 Optical micrographs of LAZ1110+Sc: (a) before rolling, showing β phase of about 36 µm and about 6.6% small α particles; (b) 30%, (c) 60% and (d) 90% rolling reduction



Fig.4 Optical micrographs of LAZ1110+Be: (a) before rolling, showing β grain of about 75µm and about 10.6% α phase distributing in Widmanstatten structure; (b) 30%, (c) 60% and (d) 90% rolling reduction



Fig.5 Optical micrographs of LAZ1110+Sc+Be: (a) before rolling, showing β phase of about 33µm and about 2.4% small α particles; (b) 30%, (c) 60% and (d) 90% rolling reduction



Fig.6 Optical micrographs of solution treated specimens: (a) LAZ1110; (b) LAZ1110+Be; (c) LAZ1110+Sc; (d) LAZ1110+Be+Sc



Fig.7 XRD patterns of solution treated specimens



Fig.8 Tensile strength of specimens solution treated at 400 °C for 30 min, then quenched in water

mechanism functions effectively, while the hot extrusion process does not. It also indicates that solution treatment endows the Mg-Li alloys with internal strain energy, if it is conserved and added up to that from cold rolling, recrystallization with fine grains is easily achieved upon subsequent annealing. So, all the four solution treated alloys are immediately cold-rolled and annealed. It is found that 60% rolling reduction and annealing at 250 °C can yield fine grains, as shown in Fig. 9.

4 Conclusions

1) A processing route to obtain fine grained LAZ1110 alloy which is basically single-phase structured was studied. The process was based on cold-rolling, which was not adopted before in preparing fine-grained Mg-Li alloys.

2) Solution treatment followed by immediate water quenching, cold rolling and annealing produced a distinct fine-grained structure, which was unprecedented by rolling Mg-Li alloys with Li content more than 11%.

3) Some minute amount (<0.02%) of Sc and/or Be were added to the LAZ1110. They showed some grain refining effect in large grain scale in the individual or intermittent processing stage. However, the final fine grain size (about 2 μ m) obtained following the above recipe was not affected by the addition of minute amount (<0.02%) of Sc and/or Be.



Fig.9 Optical micrographs of various LAZ1110 alloys: (a) LAZ1110; (b) LAZ1110+Be; (c) LAZ1110+Sc; (d) LAZ1110+Be+Sc

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