Effect of Al and Mn Content on the Mechanical Properties of Various ECAE Processed Mg-Li-Zn Alloys

Jian-Yih Wang, Tien-Chan Chang, Li-Zen Chang and Shyong Lee

1Department of Materials Science and Engineering, National Dong Hwa University, Hualien 97401, Taiwan
2Department of Mechanical Engineering, National Central University, Jhongli 32001, Taiwan

The equal channel angular extrusion (ECAE) process is an innovative method to refine grain structure; however, it could be highly technical to perform to result in subsequent exotic mechanical properties. This study will demonstrate how easy or difficult of this operation. Yoshida et al. had applied the ECAE process on Mg-10%Li-1%Zn alloy to obtain a max. superplastic elongation of 421%. This paper tries to reproduce it with Mg-11%Li-1%Zn and Mg-9%Li-1%Zn alloys via the same ECAE process. For the 28 specimens having received 4 passes of ECAE, one of them shows a comparable but lesser elongation of 350% under selective temperature of 523 K and strain rate, and the corresponding strain rate sensitivity exponent is 0.48. Furthermore, the ECAE process is imposed on three other Mg-Li-Zn alloys containing Al and Mn (Mg-9%Li-1%Zn-0.2%Mn, Mg-9%Li-1%Zn-1%Al-0.2%Mn and Mg-9%Li-3%Al-1%Zn-0.2%Mn), and this investigation is unprecedented. The original justification for this exploration is that Al and Mn addition may strengthen the phases which are the sole micro-constituents of Mg-Li alloys when bearing Li content between 5% to 11%. Indeed, it is so at room temperature as determined by micro hardness testing. However, high temperature tensile elongation is not benefited.

Keywords: magnesium-lithium-zinc alloy, equal channel angular extrusion, grain refining, superplasticity

1. Introduction

Magnesium alloy is the lightest metallic material for structural application. However, it is commonly recognized that magnesium possesses poor formability because of its hexagonal close-packed structure. To make up for this shortcoming and further reduce weight, alloying magnesium with lithium of extremely low density, 0.534 g/cm³, can achieve both goals. According to the Mg-Li phase diagram, with Li content between ~5 mass% to 11 mass%, the BCC structured β phase of Li solid solution will co-exist with the HCP α phase of Mg solid solution.1) This dual-phase structure may be beneficial for superplasticity. The simple binary Mg-8.5%Li and Mg-9%Li alloys were studied and demonstrated indeed to have high elongation of 610% and 460% respectively.2,3) Recently, Yoshida et al.4) applied the equal channel angular extrusion (ECAE) on the Mg-10.2%Li-0.8%Zn alloy aiming at super refining the grain size in order to obtain even higher superplastic elongation. Their highest elongation is 421% which is good but not exceptional. We are interested to repeat the work with some variation to see how easy is the ECAE process to be operated in modifying metals for impressive mechanical properties. In this paper, five alloys with Li content of 9 and 11 mass% plus various other elements-Zn, Al and Mn were studied. One percent addition of zinc is common among presently employed magnesium alloys, and it can dissolve in both α and β phases with the expectation of strengthening the alloys.5-7) The aluminum is common for strengthening and corrosion resistance,7) and this former effect will be verified by comparing the alloys with or without Al content.

2. Materials and Experimental Procedures

All five alloys contained one percent zinc plus various amounts of Li, Al and Mn. Their designations and analyzed compositions are listed in Table 1. The die employed for the equal channel angular extrusion (ECAE) is schematically shown in Fig. 1. The channel of the ECAE die has a square cross-section measuring 20 mm on the side and makes a 90° angular turn. The die was placed in a hot press which...
provided a 373 K, 423 K or 448 K isothermal condition as well as the external force for pushing the bar through the angular channel. The strain per pass can be significant, and can be accumulated by making several passes to achieve even higher strain if desired. In ECAE processing, the billet is often rotated around its axis between extrusion cycles to develop different microstructures. In our experiment, most specimens were rotated 180° between two consecutive passes and were designated as route C condition. Some specimens received another processing route whereby the specimens were rotated 90°, and were designated as Bc. Based on previous lab experience in ECAE process work on magnesium alloy AZ 31,8,9) four angular extrusion passes were decided as being optimum.

The tensile tests were made on an Instron machine at atmospheric and high temperatures. Other apparatuses used in the study included X-ray diffraction spectrometer, optical microscope, transmission and scanning electron microscopes. The micro hardness tests were done to determine the difference between two micro-constituents, α and β phases.

3. Results and Discussion

3.1 Microstructure and room temperature mechanical properties prior to ECAE

The basic micro-constituents were α and β phases at different ratios (Fig. 2). The X-ray diffraction analysis (Fig. 3) confirms this morphology assertion. In a previous relevant paper9) by the author, it was speculated that there were oxide particles in the α matrix, and they were identified as MgO and ZnO. The tensile strengths and elongations are plotted in Fig. 4. The alloy containing 11% Li (LZ111) is weaker than the 9% Li group. This is simply due to the higher percentage of β phase which is weaker than α. The addition of 0.2% Mn decreases the strength, while the other element, Al, can enhance the strength as demonstrated by the two alloys containing 1% and 3% Al. All five Mg-Li-Zn alloys show high elongation over 45%, which is indicative of possible good formability at room temperature.

3.2 After four passes of ECAE

After the ECAE process via Bc and C routes at 373 K,
423 K and 448 K, respectively, the strength of the Mg-Li-Zn alloys were significantly raised, accompanied by some reductions in elongation (Fig. 5). Both the \(\alpha\) and \(\beta\) phases showed positive response to ECAE process as their individual micro hardness was increased and extruded structure was created (Figs. 6, 7). The specimens after 4 passes of ECAE process on Mg-9\%Li-1\%Zn (LZ91) alloy was observed with TEM showing that the \(\alpha\) phase was refined to \(0.5\) \(\mu\)m and distributed in the \(\beta\) matrix (Fig. 8).

### 3.3 Superplasticity of the ECAE processed alloys

As listed in Table 1, two Mg-Li-Zn alloys without Al and Mn addition contains Li\% slightly above and below the material used in Yoshida’s work. So, our specimens were expected to show comparable superplastic elongation and indeed the highest elongation was 350\% as shown in Fig. 9, which was tested at 523 K and strain rate of \(1.0 \times 10^{-4}\) s\(^{-1}\). The corresponding strain-rate-sensitivity exponent, m, is 0.48. This correlation between elongation and m value quite agrees with Yoshida’s work-421\% elongation associated with the m value of 0.5. It is noted that their specimens receiving 6 passes of ECAE in contrast to our 4 passes. In these two alloys, 28 specimens were tested under various
temperature and strain rate combination, 6 yielded elongations over 200% indicating that the operations of ECAE and tensile testing are highly technical for exceptional superplasticity.

Fig. 7 Microstructures of the ECAE processed Mg-Li-Zn alloys (after 4 passes), (a) LZ111 (Bc Route, 373 K), (b) LZ91 (Bc Route, 373 K), (c) LZM910 (C Route, 373 K), (d) LZAM9110 (C Route, 423 K), (e) LAZM9310 (C Route, 448 K).

Fig. 8 TEM micro-graph after 4 passes of ECAE process on Mg-9%Li-1%Zn alloy, (a) bright field.
It was demonstrated above that adding Al and Mn enhances the strengths of Mg-Li-Zn alloys at room temperature due to solid solution strengthening of the microconstituents as verified by the micro hardness test. However, the anticipation of increasing superplasticity was not realized. The highest tensile elongations obtained were 283% and 278% with 30 tests on specimens in the three categories containing Al and Mn under various combinations of strain rate and temperature. Those specimens showing tensile elongation above 200% are listed in Table 2. Figure 10 is plotted to show the flow stress vs. strain rate of all tested specimens (58 pieces), which belong to the five categories of Mg-Li-Zn alloys. In each sub-figure, maximum strain rate sensitivity exponent exhibiting in that category is indicated. It is noted that the group showing highest index of 0.48 produces ultimate elongation of 350% and more high elongation specimens while the lowest 0.28 one shows no elongation over 200%.

4. Summary

A comprehensive study of ECAE processed magnesium-lithium-zinc alloys were conducted with a good reference by Yoshida et al. to compare with. Applying the ECAE on additional Mg-Li-Zn alloys containing Al and Mn for superplasticity consideration has not been touched before. The ECAE process is certainly effective in enhancing the strength of the processed material at room temperature. From the results of five Mg-Li-Zn alloys with grains refined by the ECAE process, Mg-9Li-1Zn alloy showed the greatest

<table>
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<tr>
<th>Materials</th>
<th>Temp. (K)</th>
<th>Strain rate (s(^{-1}))</th>
<th>Flow stress (MPa)</th>
<th>Elongation (%)</th>
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<td>208</td>
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<tr>
<td></td>
<td>523</td>
<td>1 \times 10^{-3}</td>
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<td>LZ91</td>
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</table>

Fig. 10 Flow stress vs. strain rate for various ECAE processed Mg-Li-Zn alloys as tested at selective temperatures. In each sub-graph, a maximum obtainable strain rate sensitivity exponent is indicated.
increase in tensile strength, about 41.8 MPa, and the least decrease in elongation, about 25%, with a prolonging of the α phase along the extrusion direction. After 4 passes of the ECAE process the microstructure of the Mg-9%Li-1%Zn alloy was observed by TEM indicating that the α phase was refined to ~0.5 μm and distributed in the β matrix. This fine structure may be the prime factor yielding superplastic elongation of ~350% at selective temperature and strain rate conditions.

Acknowledgment

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REFERENCES