EFFECT OF Zr ADDITION ON THE GRAIN REFINEMENT OF Mg-Li$_X$-1.5Al MAGNESIUM ALLOYS

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Abstract

Magnesium-lithium alloy is one of the lightest metal construction material and has excellent strength and with low density. Its density is about 1.3-1.6 g/cm^3 and has high strength, and is an ideal structure material for aerospace, aeronautics and military industry, nuclear industry, electronic products, etc. The primary feature of Mg-Li alloys is high solid solubility of lithium in magnesium and magnesium in lithium. There are also no intermetallic phases. In the equilibrium system, there are two solid phases: a solid solution of lithium in magnesium with a hexagonal structure and a solid solution of magnesium in the lithium, characterised by a structure of a regular spatially centred. To improve mechanical properties of magnesium-lithium alloys, a different type of modifiers are added to melts to decrease grain size thus changing mechanical properties. Zirconium is one of the magnesium alloy microstructure modifier, which reduces the grain size and consequently makes it possible to obtain satisfying mechanical properties. Even a small amount of zirconium in magnesium alloys results in a complete modification of coarse-grained microstructure. The paper presents the results of the influence of commercial Zr on the refine the grain size and dilatometric behaviour of Mg-Li$_x$-1.5Al alloys (x = 4.5, 9 and 12 by weight percentage). The coefficient of thermal expansion (CTE) of investigated light-weight alloys was measured in the temperature range from 20 to 400 °C. The effects of Zr content on the microstructure of analysed magnesium alloys were investigated. Microstructural evaluations were identified by light microscope.

Keywords: Magnesium-lithium alloys, microstructure, grain refinement, thermal expansion

1. INTRODUCTION

Magnesium alloys maintain many benefits, such as light weight, high specific strength, good vibration stability and excellent electromagnetic shielding property [1, 2]. However, magnesium alloys show the poor plastic property in the process of extrusion, rolling, forging and punching [2, 3]. To obtain components made from magnesium alloys, die-casting technology is usually used [3, 4]. Compared with the methods of deformations, this technology has disadvantages, such as low production efficiency, high production cost and relatively poor properties. Mg-Li alloys are the lightest materials with the density of 1.3-1.6 g/cm^3, however, in the engineering purpose, Mg-Li alloys still have some problems that need to be solved urgently, like relatively low strength, unstable mechanical properties, relatively high production cost [5-8]. To solve those problems, alloying is one of the commonly used methods to obtain Mg-Li base alloy with excellent properties. Al is one of the most common alloying element in Mg-Li base alloys. The solubility of Al in Mg-Li alloy is high. The addition of aluminium in Mg-Li systems mainly dissolved in solid solutions, what causes improvement of strength and increase the density, however, decrease in elongation [9-11].

The grain size is one of the most significant factors defining the property of cast metal parts causes it significantly influences on the mechanical properties. The grain size of casts can be formed by modifying many casting factors, such as the cooling rate, or by adding alloying components and nucleants (a grain refiner) previously or through the casting process. Zirconium can be added to magnesium-lithium alloys for grain refinement. One of the significant advantages received from the Zr additions is the excellent ability of grain refinement related to other processes. It is believed that zirconium, can be nucleant of magnesium-lithium alloys since of the similarity in the lattice structure, consequently giving sufficient nucleation [10-12].

In this manuscript, the influence of Zr on the microstructure, thermal stability and mechanical properties of Mg-Li base alloys are presented.

2. EXPERIMENTAL PROCEDURE

Within the framework of present work alloys of magnesium with lithium and aluminium and with Zr as grain refinement has been melt, cast and investigate. As main components used in experiments, magnesium with technical grade (min. 99.5 % Mg), aluminium 3N8 (99.98 % Al), lithium (99.9 % Li), and Zr (0.2 and 0.5 % Zr) as refinement were utilised. Melting and casting of alloys were carried out using laboratory vacuum induction furnace VSG 02 from the company Balzers [13]. Melts carried out in a crucible of Al_2O_3 in shape of ø60x80 mm, using the ceramic material sheath thermocouple for measuring the temperature of melting and casting alloys.

Magnesium, lithium and aluminium were placed directly in the crucible. Before melting, after closing the furnace, repeatedly pumped out a working chamber of the furnace, blowing with argon. As a result, it minimises the residual amount of oxygen in the chamber, which could cause contamination and oxidation of the prepared alloy. Melts were carried out in argon at a pressure of 86.7 kPA, to minimise the starting components evaporation. Melting temperature was approx. 700÷720 °C and the melting time approx. 5 min., which, taking into account the strong bath stirring electrodynamic eddy currents in enough for the complete homogenization of the melt. Grain refinement was introduced at the end of the melting process from the vacuum containers. After placed of grain refinement in the alloy, melts kept in the liquid state for 2 minutes, followed by the casting. Alloys were cast by gravity into the cold mould of graphite to give rod-shaped ingots with dimensions ø20x100 mm. Ingots were characterised by a homogeneous, fine-grained structure and not found to the occurrence of casting defects. The chemical composition of achieved alloys is presented in **Table 1**.

Table 1 Chemical composition of formed Mg-Li-Al alloys (wt.%)

Li	Al	Zr			Mg
4.5	1.5	-	0.2	0.5	Balance
9	1.5	-	0.2	0.5	Balance
12	1.5	-	0.2	0.5	Balance

The linear thermal expansion of the investigated magnesium-lithium alloys was measured in argon atmosphere using the Bahr 805A/D dilatometer over a temperature range from ambient temperature to 400 °C at heating and cooling rates of 1 K/min.

The as-cast grains of the etched samples were examined using polarised light in optical microscope Leica equipped Q-WinTM image analyser. The grain size was measured by the linear intercept method at the centre of transverse sections.

Hardness tests were made using Zwick ZHR 4150 TK hardness tester in the HRF scale.

3. RESULTS AND DISCUSSIONS

The optical micrographs of the as-cast Mg-Li-Al alloys with different level of Li and Zr content are presented in **Figure 1**. Equiaxed grains were obtained in all samples, and no columnar structure was observed.

The microstructures of specimens after casting are shown in **Figure 1**. The tested alloy Mg4.5Li1.5Al is characterised by α-Mg phase microstructure of solution Li in Mg (Mg solid solution, HCP structure). A Mg9Li1.5Al alloy is characterised with a typical duplex phase microstructure composed of a solid solution of lithium in magnesium with hexagonal system and magnesium in lithium with regular body-centered system. A Mg12Li1.5Al is characterised by a single microstructure of solution β-Li phase (Li solid solution, BCC structure).

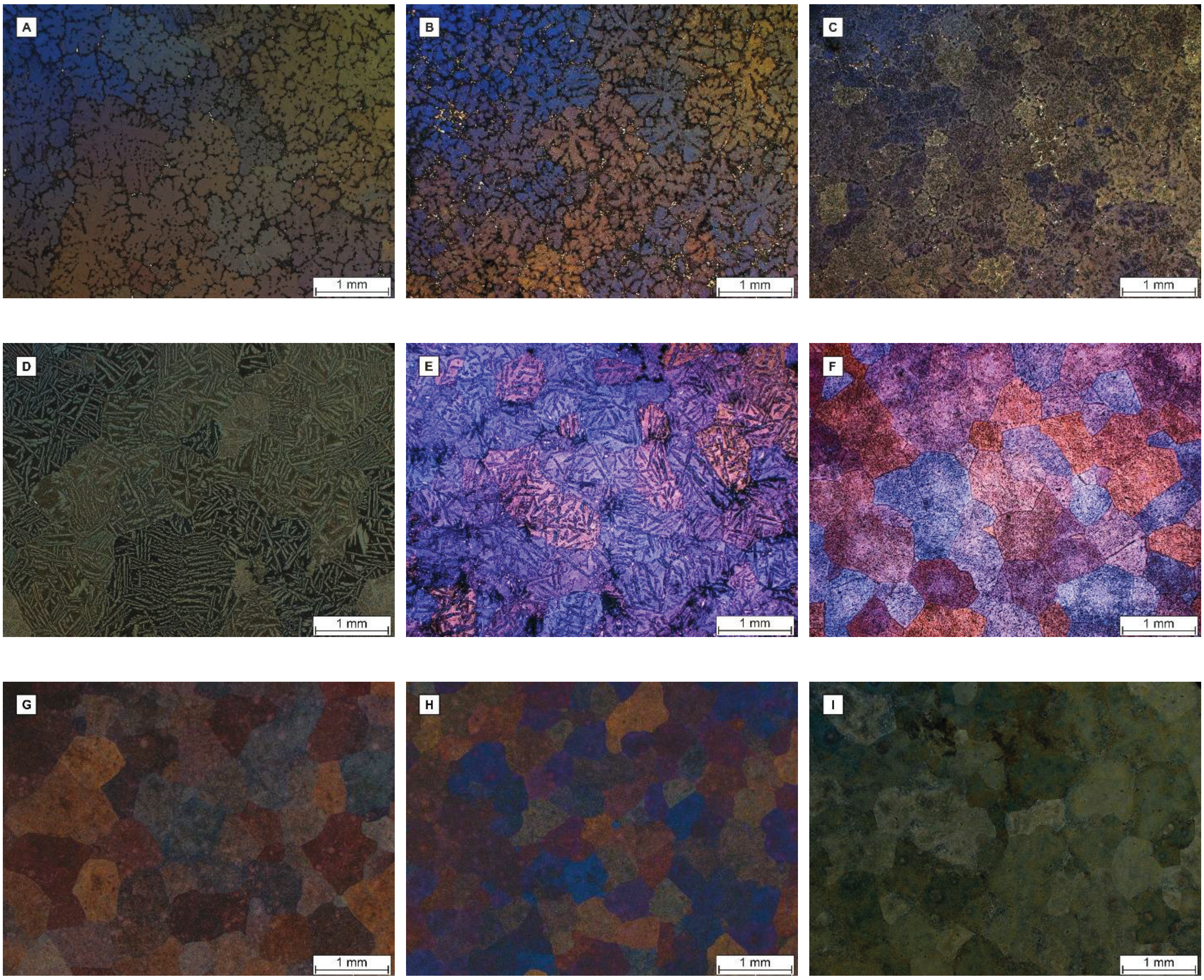

Figure 1 Optical micrographs showing the grain size of Mg-Li-Al base alloy at various level of Li and Zr (in wt.%): a) Mg-4.5Li-1.5Al, b) Mg-4.5Li-1.5Al+0.2Zr, c) Mg-4.5Li-1.5Al+0.5Zr, d) Mg-9Li-1.5Al, e) Mg-9Li-1.5Al+0.2Zr, f) Mg-9Li-1.5Al+0.5Zr, g) Mg-12Li-1.5Al, h) Mg-12Li-1.5Al+0.2Zr, i) Mg-12Li-1.5Al+0.5Zr, Nomarsky contrast

Figures 1 and **2** clearly show that the grain refining appearance is different for each alloy. Once Zr is added to every alloy there is a significant reduction in grain size and then the grain size decreases continuously with further increments in Zr. For example, the Mg4.5Li1.5Al alloy can be refined from 927 to 683 µm by 0.2 wt.% Zr, while 0.5 wt.% Zr causes decreases to 451 µm. Due to the addition of Zr, the grain size of the Mg9Li1.5Al alloy was reduced obviously. When the addition of Zr was 0.2 wt.%, the mean grain size of the Mg9Li1.5Al alloy was reduced from 789 to 630 μm. Further increasing the addition of Zr caused the grain size slightly decrease to 613 μm. It can be seen from **Figure 1** and **2** that the grain size of Mg12Li1.5Al alloy untreated by Zr, is high compared with alloys modified with Zr. However, after being treated by the 0.5 wt.% Zr, the grain size decreased from 649 μm to 461 μm.

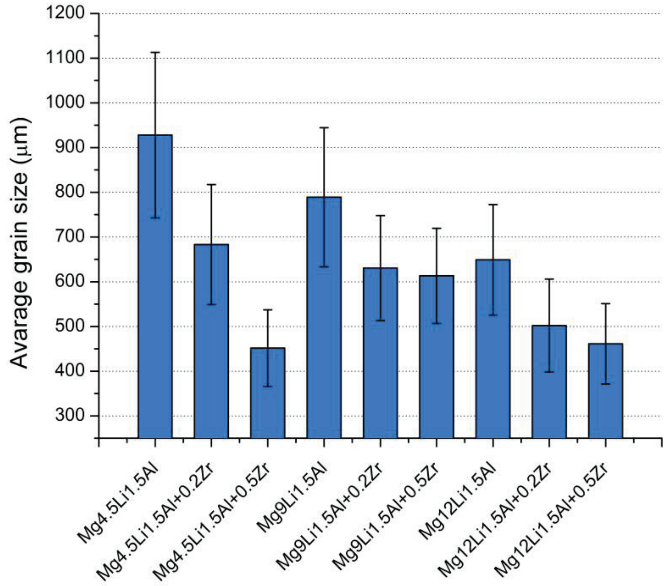

Figure 2 Variation of grain size of investigated Mg-Li-Al alloys with the addition levels of Zr

Changes in hardness were plotted against the content of Li and Zr, as presented in **Figure 3.** The hardness of unmodified Mg4.5Li1.5Al alloy was 37.2 HRF which increased to 49.9 HRF with 0.2 wt.% Zr addition. Furthermore, an increase in hardness to 57.7 HRF with Zr levels up to 0.5 wt.% was also observed. Hardness measurements of Mg12Li1.5Al with a change in the concentration of Zr from 0.2 to 0.5 wt.%, the hardness increased from 70.4 HRF to 85.6 HRF. Modification of Mg9Li1.5Al by zirconium has no influence on the hardness of analysed alloy, and it is approx. 63 HRF. Besides, it can also be observed that the hardness increases with increasing Li content. For instance, by increasing the Li content from 4.5 to 12 wt.% the hardness increases from 37.2 HRF to 70.4 HRF.

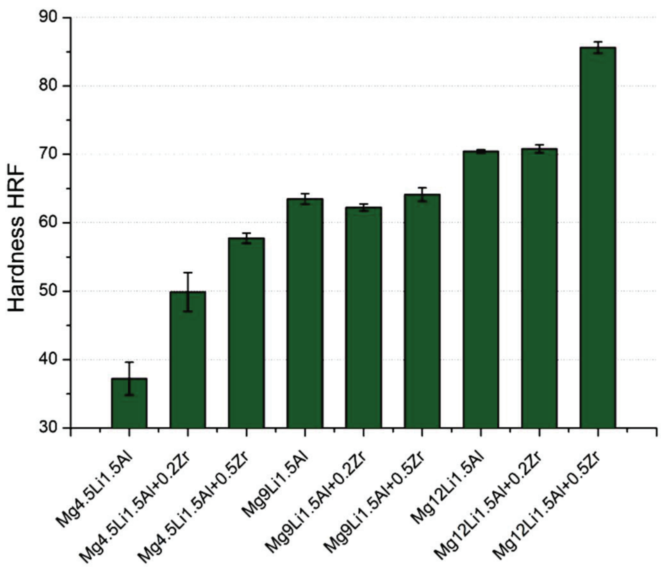

Figure 3 Hardness of investigated Mg-Li-Al alloys

Based on the data obtained from the dilatometric tests (**Figure 4)** determined coefficient of thermal expansion α (CTE). Analysis of the heating and cooling dilatometric curves of analysed materials has been found that a Mg4.5Li1.5Al alloy with HCP structures characterised by a linear increase in expansion coefficient as a function of temperature. The CTE slightly increases with an increase in temperature from 27 to $29{\cdot}10^{-6}\,K^{-1}$. Modification by zirconium has no effect on CTE in analysed alloy. The opposite situation occurs in a case of Mg12Li1.5Al alloy with BCC structure. With the increase in temperature, the value of CTE slightly decreases from 38 to $36.46{\cdot}10^{-6}\,K^{-1}$. Modification causes an increase in CTE of analysed alloy.

The analysis of CTE of Mg9Li1.5Al with duplex structure has validated the fact that linear coefficient of thermal expansion depends on temperature and content of Zr. Untreated Mg9Li1.5Al alloy has CTE about $29.4{\cdot}10^{-6}K^{-1}$, and with further increasing temperature to 250 °C CTE increases to $34.39{\cdot}10^{-6}K^{-1}$. With further increasing of temperature to 400 °C caused a decrease of CTE to $31.93{\cdot}10^{-6}\,K^{-1}$. If was also found that modification of analysed magnesium alloy causes decreases of CTE to $23.66{\cdot}10^{-6}\,K^{-1}$ at a temperature of 350 °C. Also, it can also be observed that the coefficient of thermal expansion increases with increasing Li content.

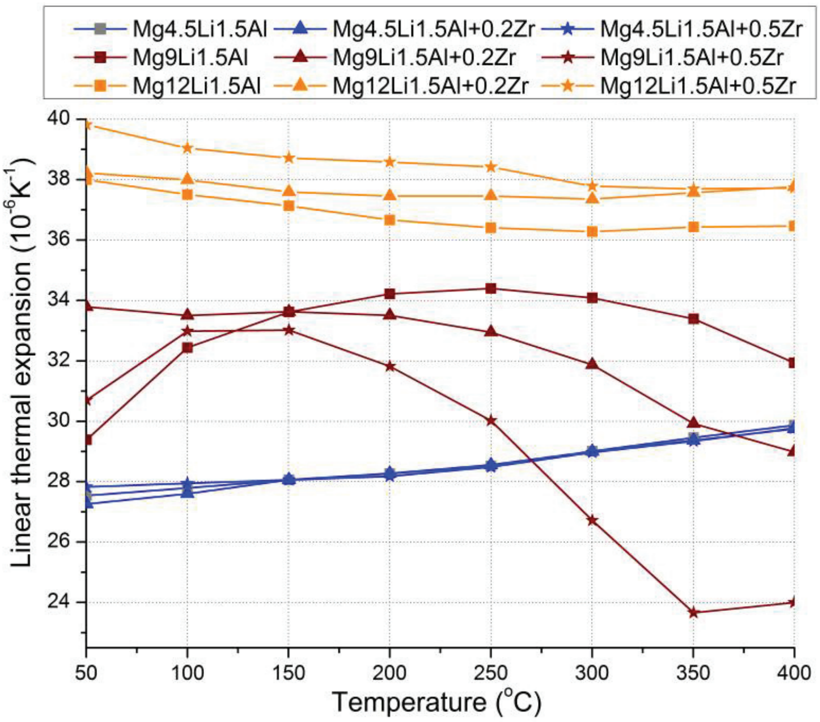

Figure 4 Changes of linear thermal expansion coefficient of investigated Mg-Li-Al alloys

4. CONCLUSION

Zirconium is an efficient grainrefiner for the Mg-Li-Al cast alloys. With the increase of the addition of Zr, the grain size of Mg4.5Li1.5Al cast alloy is reduced obviously, and the smallest grain size is 451 µm when the addition of Zr is 0.5 wt.%. The grain size of Mg9Li1.5Al cast alloy is reduced to 613 µm when the addition of Zr is 0.5 wt.%. The grain size of Mg12Li1.5Al cast alloy is reduced to 461 µm when the addition of Zr is 0.5 wt.%.

Zr has an influence on hardness for the Mg4.5Li1.5Al and Mg12Li1.5Al cast alloys. With the increase of the addition of Zr, the hardness of Mg4.5Li1.5Al increases obviously, and the highest hardness is 57.7 HRF when the addition of Zr is 0.5 wt.%. The hardness of Mg12Li1.5Al is increased to 85.6 HRF when the addition of Zr is 0.5 wt.%. The Zr addition has basically no improvement of the hardness of the Mg9Li1.5Al alloy.

Research based on dilatometer tests demonstrated that the analysed Mg4.5Li1.5Al with HCP structure and Mg12Li1.5Al with BBC structure are characterised by a linear increase and a decrease of CTE, respectively. Moreover, these two phases have different values of coefficient of thermal expansion. It was found that the coefficient of thermal expansion increases with increasing content of lithium.

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