# INFLUENCE OF EXTRUSION RATIO ON MICROSTRUCTURE AND MECHANICAL BEHAVIOR OF MG-9LI-3AL-2.5SR ALLOY

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1. College of Materials Science and Engineering, Chongqing University, Chongqing, China 2. National Engineering Research Center for Magnesium Alloys, Chongqing 400044, China **Abstract:** The as-cast Mg-9Li-3Al-2.5Sr alloy ingots were prepared and the extrusion of as-cast Mg-9Li-3Al-2.5Sr alloy ingots was carried out at 573 K (300 °C) with different extrusion ratio. The microstructure evolution during extrusion and the mechanical response of the extruded Mg-9Li-3Al-2.5Sr alloy are discussed and reported. The results show that both as-cast and extruded Mg-9Li-3Al-2.5Sr alloys contain h.c.p.  $\alpha$ -Mg, b.c.c.  $\beta$ -Li and Al<sub>4</sub>Sr phase. With the increase of extrusion ratio, deformation induced phase transformation may occur and part of h.c.p.  $\alpha$ -Mg phase transforms into b.c.c.  $\beta$ -Li phase during the extrusion. The strength of Mg-9Li-3Al-2.5Sr alloys decreases whereas ductility increases with the increasing extrusion ratio as a result of phase transformation.

Keywords: Extrusion ratio; Microstructure; Mechanical properties; Mg-Li alloy;

## **1** Introduction

The demand for lightweight metallic materials is fueled in part by the strategic need to reduce use of fossil fuels while minimizing environmental impact. Under these constraints, Mg emerges as an attractive metal system, given its low density and attractive combination of physical and mechanical attributes. Therefore, considerable attention has been focused on Mg alloys [1, 2]. Mg-Li alloys with low density, high specific stiffness, and good ductility are the lightest known metallic structural materials[3-5]. Mg-Li alloys have been used in the electrical, defense and aerospace industries[6]. The Li content in Mg-Li alloys is a key factor that influences both microstructure and mechanical response. According to Mg-Li phase diagram[7], when Li contents < 5.5 wt.%, Mg-Li alloy is comprised of a single  $\alpha$ -Mg phase with hcp structure which is Mg solid solution formed by Li dissolving in Mg. Alternatively, when 5.5 <wt.% Li < 11.5, a duplex alloy with eutectic microstructure is formed containing both hcp structured  $\alpha$ -Mg phase and bcc structured  $\beta$ -Li phase which is Li solid solution formed by Mg dissolving in Li. When wt.% Li > 11.5%, the corresponding

alloy consists exclusively of the bcc  $\beta$ -Li phase[8, 9].

Mg-Li alloys with eutectic composition posses a good combination of ductility and strength. Thus Mg-Li alloys with eutectic composition have strong potential for application. Previous study shows that adding 2.5wt% Sr in Mg-9Li-3Al alloys can improve the mechanical properties of this alloy. Mg-9Li-3Al-2.5Sr alloy has good mechanical properties with a tensile strength of 235 MPa, yield strength of 221 MPa and the elongation of 19.4% [10]<sup>-</sup> Gain refinement via plastic deformation methods represents a recently studied approach to achieve improved strength in Mg-Li alloys. Deformation methods such as extrusion, rolling, and forging, have been used to further strengthen Mg-Li alloys[11]. However, review of published literature, there are no reports about the influence of extrusion parameter on the microstructure and mechanical behavior of Mg-9Li-3Al-2.5Sr alloy. Thus, the influences of extrusion ratio on the microstructure and mechanical behavior of Mg-9Li-3Al-2.5Sr alloy are investigated in this research. The microstructure evolution during the extrusion is discussed.

## 2 Experimental

Mg-9Li-3Al-2.5Sr alloy is focused in this study. The raw materials used in this study are pure Mg (99.9 wt.%), pure Li (99.9 wt.%) and a Mg-8Sr master alloy (Mg 92 wt.%, Sr 8 wt.%). Target alloy was melted in vacuum melting furnace under the protection of argon atmosphere. Then the melt was cast in a permanent mold  $\Phi$ 90×300 mm to obtain as-cast specimens. Chemical composition of the alloy was measured by atomic absorption spectrometry (aas) using a Hitachi Z-8000 atomic absorption spectrophotometer and the alloy is composed of 8.56 wt% Li, 3.12 wt% Al, 2.47wt% Sr with the balance Mg. Homogenizing treatment was carried out at 533 K (260 °C) for 12 h in a vacuum furnace. Then the as-cast specimens were machined from  $\Phi$ 90 mm to  $\Phi$ 80 mm. The specimens were extruded at 573 K (300°C) from  $\Phi$ 80 mm to  $\Phi$ 25mm,  $\Phi$ 15mm and  $\Phi$ 9mm with an extrusion ratio of 10, 28 and 79 respectively.

The microstructure of the as-cast and extruded samples was studied using JEOL JSM 6460LV SEM following standard sample preparation procedures and the etchant used was 2vol% HNO<sub>3</sub> alcohol solution. The SEM images were acquired using secondary electrons (SE) detector. Phase analysis of the test alloys was conducted using x-ray diffractionon a D/MAX-2500pc diffractometer. TEM and STEM observations were carried out on a JEOL 2500SE TEM/STEM microscope operating at 200 kV.

Tensile specimens with gauge dimensions of  $30 \times \Phi6$  mm were machined from the central regions of the extruded alloys. Tensile tests were performed on a CMT-5105 (SANS Materials Analysis Inc., Shenzhen, China) tensile tester with a displacement speed of 1 mm/min.

## **3** Results and discussion

#### 3.1 Microstructure and phase analysis of Mg-9Li-3Al-2.5Sr alloys



Figure 1. SEM morphology and XRD pattern of as-cast Mg-9Li-3Al-2.5Sr alloy (a) SEM morphology; (b) XRD pattern.

The SEM morphology and XRD pattern of as-cast Mg-9Li-3Al-2.5Sr alloys is shown in Figure 1. The SEM micrograph and XRD pattern of as-cast Mg-9Li-3Al-2.5Sr alloy shows that LAJ932 alloys contain  $\alpha$ -Mg phase with hcp lattice structure,  $\beta$ -Li phase with bcc lattice structure and fish-bone like intermetallic Al<sub>4</sub>Sr phase.

Figure 2 shows the microstructure vertical to the extrusion direction while figure 3 shows the microstructure along the extrusion direction of extruded Mg-9Li-3Al-2.5Sr alloys with different extrusion ratio at extrusion temperature of 300°C. Figure 2 and figure 3 represent that the extruded Mg-9Li-3Al-2.5Sr alloys consist of  $\alpha$ -Mg phase,  $\beta$ -Li phase and Al<sub>4</sub>Sr phase. The grains of the extruded alloy is much finer than the as-cast the alloy. The grain size of the extruded Mg-9Li-3Al-2.5Sr alloy decreases with the increase of extrusion ratio. The dark area in Fig 3 (a)(c)(e) shows the intermetallic phase, the grey area is the  $\beta$ -Li phase while bright area is the  $\alpha$ -Mg phase. The grey area increases with the increase of extrusion ratio which means that the content of  $\beta$ -Li phase increases with the increase of



Figure 2. SEM morphology of extruded LAJ932 alloy with different extrusion ratio at extrusion temperature of 300°C (vertical to the extrusion direction): (a)(b) 79; (c)(d) 28; (e)(f) 10.

extrusion ratio.



Figure 3. Microstructure of extruded LAJ932 alloy with different extrusion ratio at extrusion temperature of 300°C (vertical to the extrusion direction): (a)(b) 79; (c)(d) 28; (e)(f) 10.



Figure 4. STEM images showing Al<sub>4</sub>Sr phase



Figure 5. XRD patterns of extruded Mg-9Li-3Al-2.5Sr alloys with different extrusion at extrusion temperature of 300°C

Figure 4 is the STEM images showing the intermetallic phase in the extruded alloy. The continuous fish-bone like  $Al_4Sr$  phase in the as-cast Mg-9Li-3Al-2.5Sr alloy is fractured and the distribution of  $Al_4Sr$  phase is improved a lot by extrusion.

Figure 5 shows the XRD patterns of extruded Mg-9Li-3Al-2.5Sr alloy with different extrusion ratio at extrusion temperature of 300°C. The XRD results confirm that both the as-cast and the extruded Mg-9Li-3Al-2.5Sr alloys are composed of  $\alpha$ -Mg,  $\beta$ -Li and Al<sub>4</sub>Sr phase. The intensity of  $\alpha$ -Mg phase peaks and  $\beta$ -Li phase peaks changes with the change of extrusion ratio. The relative intensity of  $\beta$ -Li phase peaks increases with increasing extrusion ratio.

#### 3.2 Microstructure evolution during the hot deformation

Generally, the relative content of  $\alpha$ -Mg and  $\beta$ -Li in Mg-9Li-3Al-2.5Sr alloys can be estimated based on the integrated intensity of the observed peaks in the diffraction patterns. The equation as follows is used to estimate the relative content of  $\alpha$ -Mg and  $\beta$ -Li phase.

$$\frac{X_{\alpha}}{X_{\beta}} = \frac{I_{i,\alpha}}{I_{j\beta}} \cdot \frac{I_{j,\beta}^{Fa}}{I_{i,\alpha}^{Fa}} \cdot \frac{RIR_{\beta,c}}{RIR_{\alpha,c}}$$
(1)

where  $X_{\alpha}$  and  $X_{\beta}$  are the weight fractions of  $\alpha$ -Mg phase and  $\beta$ -Li phase in the alloy respectively.  $I_{i,\alpha}$  is the integrated intensity of peak I of  $\alpha$ -Mg phase, and  $I_{j,\beta}$  is the integrated intensity of peak j of the  $\beta$ -Li phase in the alloy, which are obtained from the XRD experiment data.  $I_{i,\alpha}^{rel}$  is the intensity of peak i of  $\alpha$ -Mg phase relative to the intensity of the strongest peak in  $\alpha$ -Mg phase while  $I_{j,\beta}^{rel}$  is the intensity of peak j of  $\beta$ -Li phase relative to the intensity of the strongest peak in  $\beta$ -Li phase, which can be extracted from PDF cards. The values of RIR<sub> $\beta,c</sub>$  and RIR<sub> $\alpha,c</sub>$  are 0.77 and 2 respectively gotten from the XRD cards. The relative content of  $\alpha$ -Mg and  $\beta$ -Li phase is based on the averages of values that calculated using three different peaks in order to reduce the influence of the experiment condition and specimen condition. The XRD patterns of the extrude Mg-9Li-3Al-2.5Sr alloys shows that the intensity of  $\beta$ -Li phase peaks increases with the increase of extrusion ratio. According to equation 1, the Li content increases. This result also is consistent with the microstructure observation.</sub></sub>

The result indicates that phase transformation that part of h.c.p.  $\alpha$ -Mg phase transformed into b.c.c.  $\beta$ -Li phase occurs during the extrusion. Deformation induced phase transformation often happens in steel and TiAl alloys. Usually, the transformation is dependent on the deformation temperature, strain and grain size [12]. For two phase  $\alpha$ (h.c.p)+ $\beta$ (b.c.c) Mg-9Li-3Al-2.5Sr alloy, deformation initially takes place in  $\beta$ -Li phase due to its good ductility at the first stage of extrusion. With the increase of strain, the dislocations are piled up at the  $\alpha$ -Mg/ $\beta$ -Li interface and the Gibbs free energy in the alloy may increase, which supply the drive force for the phase transformation. Strong stress concentration during deformation plays an important role in the occurrence of phase transformation. Therefore, the nucleation of  $\beta$ -Li phase may predominantly occur at the  $\alpha$ -Mg/ $\beta$ -Li interface. Figure 6 is the TEM images in the extruded Mg-9Li-3Al-2.5Sr alloys with extrusion ratio of 28. The image shows that the nucleation of  $\beta$ -Li grains occurs at  $\alpha$ -Mg/ $\beta$ -Li interface. However, the



Figure 6. TEM images showing  $\alpha$ -Mg grains ,  $\beta$ -Li grains and their boundaries in the extruded Mg-9Li-3Al-2.5Sr alloy with extrusion ratio of 28.

transformation mechanism which need to further study is not clear yet.

#### 3.3 Mechanical properties of Mg-9Li-3Al-2.5Sr alloys with different extrusion ratio

Figure 7 presents the typical tensile test curves of extruded Mg-9Li-3Al-2.5Sr alloys with different extrusion ratio. With the increase of extrusion ratio, the strength of alloys decreases whereas the elongation increases. When the extrusion ratio is 10, the extruded alloy has the biggest ultimate strength of 240MPa and the smallest elongation of 9.6%. When the extrusion ratio is 28, the alloy possesses an ultimate strength and elongation of 223MPa, 19.7% respectively. The extruded Mg-9Li-3Al-2.5Sr alloys with an extrusion ratio of 79 possesses the biggest elongation of 27.1%, however the strength is only 189MPa.

The previous study shows that the grains of the alloy decreases with the increase of extrusion

temperature. If one only considers grain boundary strengthening, the strength should be improved with the increasing of extrusion ratio. However, our previous research also shows that  $\beta$ -Li phase content increases with the increasing of extrusion ratio and strain induced phase transformation may exist in the alloy. The  $\beta$ -Li phase with a bcc structure possesses a lower microhardness than hcp  $\alpha$ -Mg [13-14].  $\beta$ -Li has five {110}<-111> independent slip systems whereas  $\alpha$ -Mg only has two {0001}<11-20> independent basal slip systems that are easy to activate at room temperature. Moreover, cross slip in bcc  $\beta$ -Li phase is also easy to activate [15]. Not surprisingly,  $\beta$ -Li has better ductility than  $\alpha$ -Mg; hence,  $\beta$ -Li is beneficial to ductility whereas  $\alpha$ -Mg contributes to strength. Therefore, with the increase of  $\beta$ -Li content, the ductility will be improved whereas the strength will decreases, which is consistent with our experimental results.



Figure 7. Tensile test curves of extruded LAJ932 alloy with different extrusion ratio at extrusion temperature of 300°C

## 4 Conclusions

- Both the as-cast and extruded Mg-9Li-3Al-2.5Sr alloys contain α-Mg phase, β-Li phase and Al<sub>4</sub>Sr phase. The grains of the extruded alloy are much finer than the as-cast alloy. The grain size of the extruded alloys decreases with the increase of extrusion ratio.
- 2) β-Li area and the relative intensity of β-Li XRD peaks in the extruded Mg-9Li-3Al-2.5Sr

alloy increases with the increase of extrusion ratio which means that the content of  $\beta$ -Li increases with the increase of extrusion ratio. Deformation induced phase transformation may occur during the hot extrusion. Part of h.c.p.  $\alpha$ -Mg phase transformed into b.c.c.  $\beta$ -Li phase.

- {10-10}<10-10> texture is formed in the h.c.p. α-Mg phase while {110}<101> texture is formed in the b.c.c. β-Li phase during the extrusion.
- 4) The strength of extruded Mg-9Li-3Al-2.5Sr alloys decreases whereas the ductility increases with the increase of extrusion ratio. The extruded Mg-9Li-3Al-2.5Sr alloy with extrusion ratio of 79 possesses the biggest elongation of 27.1 with strength of 189MPa. When the extrusion ratio is 10, the extruded alloy has the biggest ultimate strength of 240MPa with an elongation of 9.6%.

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