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Research Article

A First Report On Static Grain Growth Kinetics In An Ultralight Mg-8.41li-1.81al-1.77zn Alloy Subjected To Friction Stir Processing

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Abstract

Friction stir processing is one of the severe plastic deformation methods. Static grain growth study during friction stir processing does not receive enough attention compared to dynamic recrystallization study. Thus, in this report on static grain growth kinetics of Mg-Li alloy, an ultralight Mg-8.41Li-1.80Al-1.77Zn alloy has been fabricated by rolling, friction stir processing, and annealing. Microstructural examination of the nugget zone in the annealed state revealed that the grain growth rates at 523 and 573 K are much lower than the growth rate at 623 K. In the meantime, grain growth prior to 30 min is not obvious, but grains grow obviously with the increase in time after 30 min. The nugget zone grain growth kinetics equation abided by parabolic relation based on the linear fitting of the experimental grain sizes. The grain growth activation energy was 176.191 kJ/mol, higher than the lattice diffusion activation energy of magnesium, 135 kJ/mol. Probable cause is that the second phase particles increase the difficulty of thermal activation and raise the activation energy. The calculation error between theoretical grain growth model and experimental grain growth model is two orders of magnitude because of the use of an effective diffusivity. Hence, accurate theoretical model for static grain growth remains to be established in the future. This indirectly demonstrates the scientific meaning and value of established experimental parabolic growth model.

Keywords: Magnesium-lithium alloy; Friction stir processing; Annealing; Static grain growth; Microstructure

Introduction

Mg-Li alloy is the lightest nontoxic metallic alloy. Due to extremely low density, excellent specific stiffness, good specific strength, good damping property, and electromagnetic shielding performance, Mg-Li alloy has the potential for applications in aerospace, military weapons, 3C products, and automobile manufacturing fields. Thus, study on Mg-Li alloy has drawn much attention from extensive researchers [1-7]. Not only room temperature mechanical properties, corrosion performance, and microstructures [1,4,5,6] but also high temperature behavior and microstructure [2,3,7] were investigated. Because of good comprehensive mechanical properties of two-phase Mg-Li alloys, we designed an Mg-8Li-2Al-2Zn (designated as LAZ822) alloy. The purpose of 8 wt. %Li addition is to obtain a two-phase alloy. The purpose of 2 wt.% Al addition is to achieve solid solution strengthening and second phase strengthening. The purpose of 2 wt.% Zn addition is to achieve solid solution strengthening. Friction stir processing (FSP) is further development of friction stir welding and one of the severe plastic deformations (SPD) approaches. According to literature survey, FSP of Mg-Li alloys has been reported in several alloys [8-10]. The research aspect includes ambient mechanical properties, corrosion behavior, and microstructures [8] and high temperature mechanical properties and microstructures [9-10] during FSP. Thermal stability issue is often accompanied by SPD process. When the ultrafine-grained and fine-grained alloys are exposed to high temperature, grain coarsening often occurs. Some grain growth reports and review containing modelling and experimental microstructures were documented [11-14]. To the best of our knowledge, no information has been known about the study on the static grain growth behavior in Mg-Li alloy. Thus, it is necessary to work out this report on static grain growth in Mg-Li alloy. In this work. our investigated contents include four aspects: (i) to fabricate LAZ822 alloy by casting, rolling, FSP, annealing; (ii) to investigate or characterize its annealing microstructure; (iii) to investigate its grain growth kinetics; (iv) to compare established experimental grain growth equation and theoretical grain growth equation. It is hoped that this first report about static grain growth in Mg-Li alloy processed by FSP stimulates the interests of extensive Mg-Li researchers.

Experimental Procedures

The present LAZ822 alloy ingot was obtained by melting and casting process similar to the process of Mg-9.3Li-1.79Al-1.61Zn alloy ingot, as shown elsewhere in detail [15]. The analyzed chemical composition of this alloy was 8.41 mass % Li, 1.80 mass % Al, 1.77 mass % Zn and balanced Mg. The ingot was homogenized at 473 K for 20 h. After milling of the ingot surfaces, the billet with the dimensions of 24 mm×90 mm×170 mm was held at 573 K for 2 h. Then the billet was hot rolled at 573 K for 12 passes to 6 mm thickness with a percent reduction of 75%. The schematic diagram of FSP principle was shown in [10]. A rotating tool was inserted into the plate to cause intense plastic deformation at elevated temperature and achieve microstructural modification. FSP was conducted on the hot rolled plate. Water spray cooling in the working position of the plate was the cooling mode. The diameter of conical pin was 8 mm. The rotational speed of the stirring head was 800 rpm. The transverse speed of the stirring head was 45 mm/min. The rolled samples were annealed at 523, 573, and 623 K for 10-200 min. To observe the optical microstructure, the specimens were mechanically ground and polished by abrasive papers from 100# to 3000#. The etched solution was 10% HCl+90% alcohol. After etched, the specimens were rinsed by alcohol and dried by a hair drier. Olympus DSX500 optical microscope was used for the examination. Grain sizes were measured by Image-Pro Plus (IPP) software to characterize the microstructures.

Results and Discussion

Experimental static grain growth after FSP (Figures 1,2, 3) display the nugget zone microstructures of FSP LAZ822 alloy annealed at temperatures of 523, 573, and 623 K for holding times of 10, 30, and 60 min. The white phase is hexagonal closed-packed (HCP) structured α-Mg solid solution phase while the yellow phase is body-centered cubic (BCC) structured β-Li solid solution phase. Hence, this alloy is a two-phase alloy. It is noted that grain size increases with the rise in temperature. This is because increased temperature accelerates atomic diffusion and boundary migration, and boundary migration results in grain growth. In particular, the grain sizes at 573 and 623 K differ greatly, indicating that the grain size is very sensitive to the temperature. (Figure 4) presents the nugget zone microstructures of FSP LAZ822 alloy annealed at 623 K for different holding times. It is clear that grains grow with the increase in holding time. Table 1 presents the grain sizes after annealing at different temperatures and holding times. According to (Figures 1-4) and Table 1, grain size is very sensitive to annealing temperature and holding time. (Figure 5) presents the variation in grain sizes of LAZ822 alloy with different temperatures and times. It is seen that grain size increases with the temperatures. The growth rates, the slope of the curves, at 523 and 573 K are much lower than the growth rate at 623 K. In the meantime, grain growth prior to 30 min is not obvious, but grains grow obviously with the increase in time after 30 min.

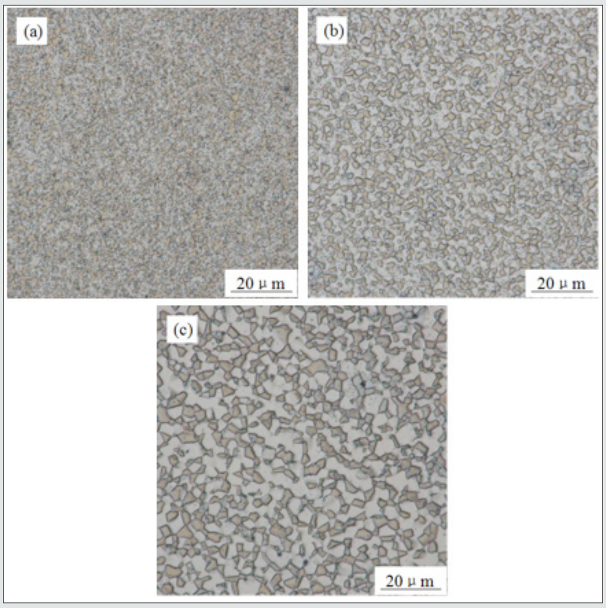

Figure 1: Nugget zone microstructures of FSP LAZ822 alloy annealed at different temperature for holding time of 10 min: (a) 523K; (b) 573K; (c) 623K.

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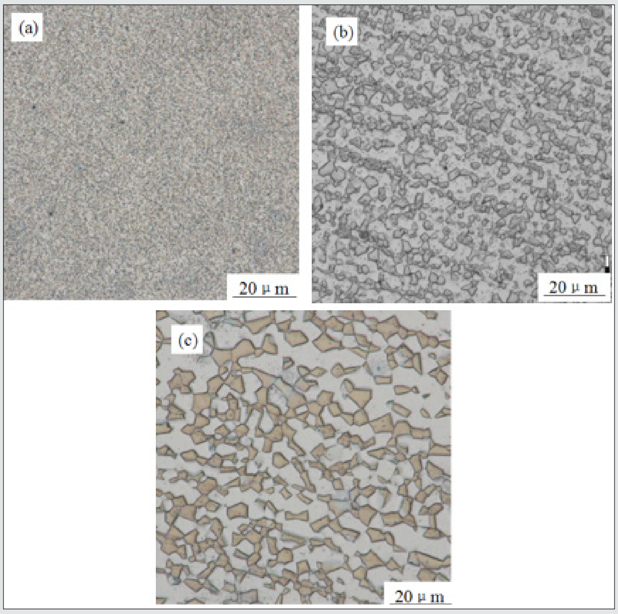

Figure 2: Nugget zone microstructures of FSP LAZ822 alloy annealed at different temperature for holding time of 30 min: (a) 523K; (b) 573K; (c) 623K.

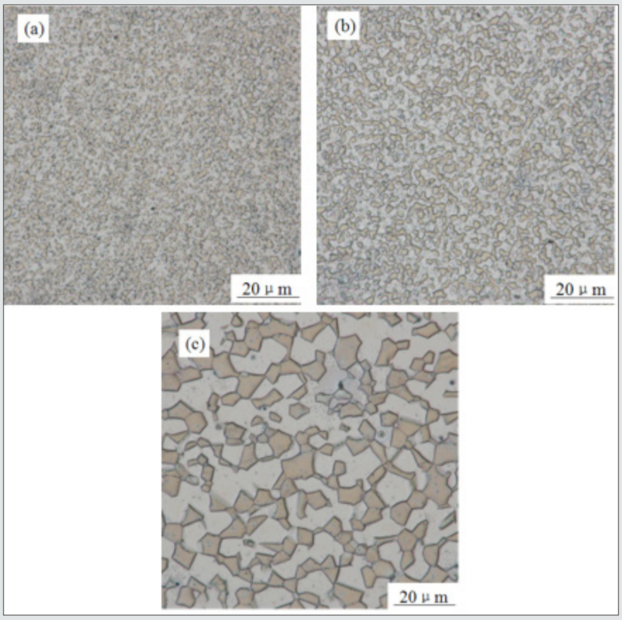

Figure 3: Nugget zone microstructures of FSP LAZ822 alloy annealed at different temperature for holding time of 60 min: (a) 523K; (b) 573K; (c) 623K.

Table 1: Grain sizes after annealing at different temperatures and holding times.

	10	30	60	100	150	200
523 K	1.19	1.62	2.22	2.67	3.05	3.28
573 K	1.52	2.5	3.45	4.32	5.11	5.55
623 K	2.79	4.84	6.79	8.87	10.78	12.6

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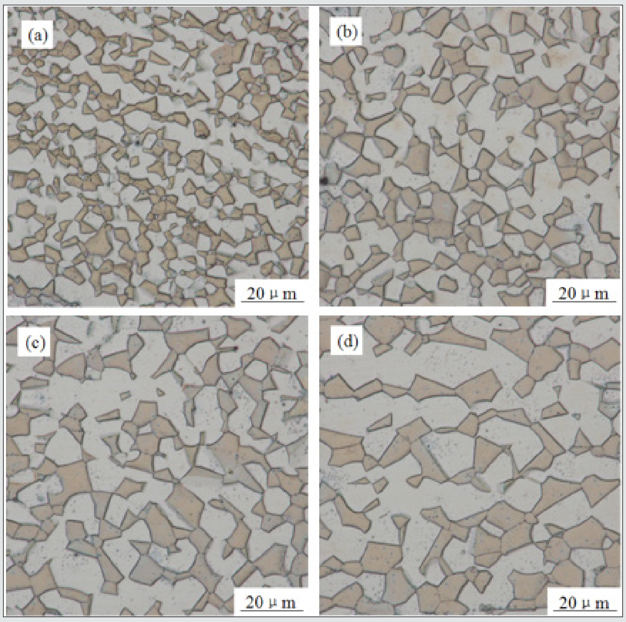

Figure 4: Nugget zone microstructures of FSP LAZ822 alloy annealed at 623 K for different holding times: (a)30 min; (b)60 min; (c)100 min; (d)150 min.

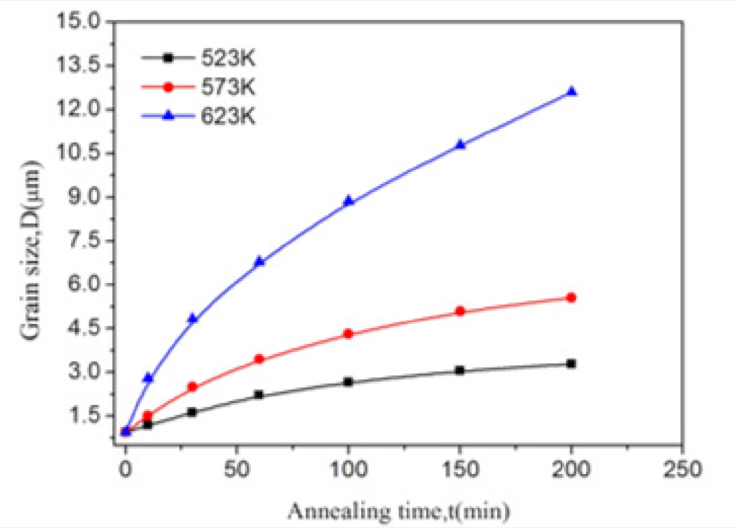

Figure 5: Variation in grain sizes of LAZ822 alloy with different temperatures and times.

Establishment of Grain Growth Kinetics Models According To Experimental Data

Static grain growth kinetics model is given by the following form:

$$D_q - D_0^q = kt \quad (1)$$

where D and D_0 are the average grain size after holding time, t, and initial grain size before annealing, respectively; $D^n - D_0^n = kt$ k is the grain growth factor, $D^n - D_0^n = kt$ and q is the grain growth exponent, relevant to the mechanism controlling the grain growth.

Differentiating Eq. (1) by time, t, one gets

$$In(\frac{dD}{dt}) = In(\frac{k}{q}) - (n-1)InD \quad (2)$$

According to Table 1, ln (dD/dt)-ln (D) curves can be plotted, and hence k and q can be obtained. (Figure 6) presents the ln (dD/dt)-ln (D) curves of FSP and annealed LAZ822 alloy. q values at 523, 573, and 623 K are equal to 2. Thus, the grain growth abides by parabolic curve. Generally, q values are 2,3,4,5, and 6 for conventional alloys. Large q values mean lower growth rate. In the present alloy, q =2 means pronounced grain growth.

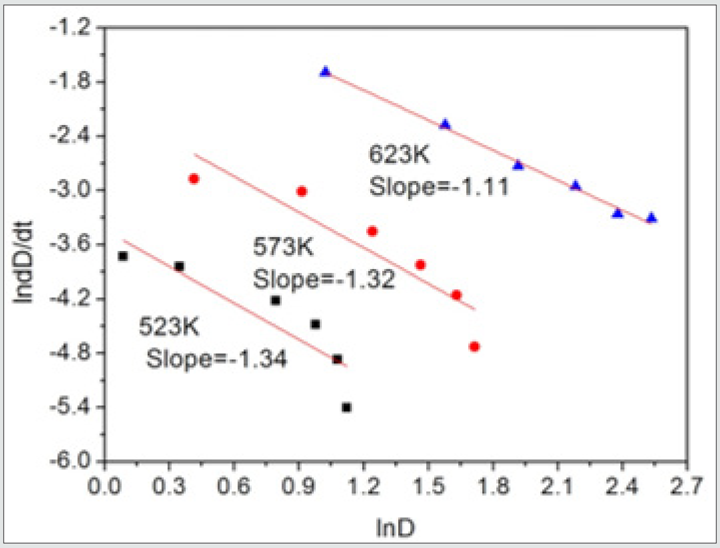

Figure 6: ln (dD/dt)-ln (D) curves of FSP and annealed LAZ822 alloy.

Thus, the static grain growth models were established as follows:

$$D^2 - D_0^2 = kt \quad (3)$$

where k values are 0.075, 0.299, and 3.69 μm^2/min at 523, 573, and 623 K, respectively.

k value is given by

$$k = k_0 \exp(\frac{-Q_g}{RT}) \quad (4)$$

where k_0 is a constant, R is the universal gas constant, 8.314 J/mol•K, and T is the absolute temperature in Kelvin.

(Figure 7) presents the $\ln k$ -1000/T curve of FSP and annealed LAZ822 alloy. The slope of fitted curve is 21.192. Thus, the grain growth activation energy, Q_g, is calculated as the following: Q_g=21.192×8.314×1000=176.191 kJ/mol. This experimental grain growth activation energy of 176.191 kJ/mol is higher than the lattice diffusion activation energy of magnesium, 135 kJ/mol [16]. Probable cause is that the second phase particles obstruct the movement of grains and increase the difficulty of softening such as annealing after FSP. In other words, the second phase particles increase the difficulty of thermal activation and raise the activation energy.

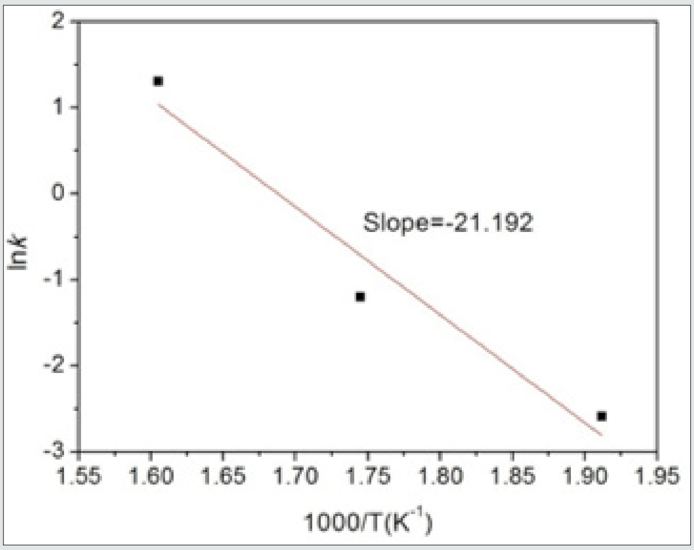

Figure 7: 1000/T curve of FSP and annealed LAZ822 alloy.

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Analysis and Comparison Of Theoretical And Experimental Static Grain Growth Equations

Theoretical static grain growth model for parabolic growth curve is given by [17]

$$d2 - d_0^2 = \left(\frac{4\gamma\Omega}{W}\right)\left(\frac{D_{gb}}{kgT}\right)t = kt \quad (5)$$

where d is the grain size after holding time of t; d_0 is the initial grain size for t=0 s; γ is the grain boundary surface tension; Ω is the atomic volume; W is the grain boundary width, W=2b, here, b is the magnitude of Burgers vector; D_{gb} is the grain boundary diffusivity; k_B is Boltzmann's constant; k is the growth factor.

An estimation is made for 573 K × 60 min situation. T=573 K, t=3600 s, d=3.45 μm, d_0=1.52 μm, γ=1 J m^{-2} [18], $b=3.21\times10^{-10}$ m [19] ,$\Omega=0.7b^3=2.315\times10^{-29}$ m^3, $D_{gb}=6.34\times10^{-11}$ m^2 s^{-1} [20], k_B $=1.38\times10^{-23}$ J K^{-1}. The theoretical k value is 1.16×10^{-9} m^2 s^{-1} while the experimental k value is 2.66×10^{-15} m^2 s^{-1}. This indicate a big diffrerence. This is because the diffusion process is not a grain boundary diffusion process but a lattice diffusion dominated process. Further estimation is made using an effective diffusivity in [21]. $D_{eff}=D_L+(W/d)D_{gb}$, where D_{eff} is the effective diffusivity and D_L is the lattice diffusivity. $D_{L=}4.27\times10^{-15}$ m^2 s^{-1} according to our previous model [20]. The theoretical k value is 2.93×10^{-13} m^2 s^{-1} while the experimental k value is 2.66×10^{-15} m^2 s^{-1}. The calculation error between theortical model and experimental model becomes a little bit closer and is two orders of magnitude because of the use of an effective diffusivity. Hence, accurate theoretical model for static grain growth remains to be established in the future. As Atkinson [22] and Baldan [23] pointed out in their articls published in 1988 and 2002, respectively, that an unified accurate grain growth model still was not avaliable although much effort has been made across the world. Our calculated results confirm what they said. This indirectly demonstrates the scientific meaning and value of established experimental parabolic growth model.

Conclusion

a. An ultralight Mg-8.41Li-1.80Al-1.77Zn alloy has been fabricated by rolling, friction stir processing, and annealing. Microstructural examination of the nugget zone in the annealed state revealed that the grain growth rates at 523 and 573 K are much lower than the growth rate at 623 K. In the meantime, grain growth prior to 30 min is not obvious, but grains grow obviously with the increase in time after 30 min.

b. The nugget zone grain growth kinetics equation abided by parabolic relation based on the linear fitting of the experimental grain sizes. The grain growth activation energy was 176.191 kJ/mol, higher than the lattice diffusion activation energy of magnesium, 135 kJ/mol. Probable cause is that the second phase particles increase the difficulty of thermal activation and raise the activation energy.

c. The calculation error between theortical grain growth model and experimental grain growth model is two orders of magnitude because of the use of an effective diffusivity. Thus, accurate theoretical model for static grain growth remains to be established in the future. This indirectly demonstrates the scientific meaning and value of established experimental parabolic growth model.

Fund Projects

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