Fabrication of Al-Coated Mg–Li Alloy Sheet and Investigation of Its Properties

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Received: 5 April 2018 / Revised: 8 May 2018 / Published online: 26 June 2018
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Abstract

An Al-coated Mg–8 mass% Li alloy rectangular bar was fabricated by hot extrusion, and then, it was hot-rolled into a thin sheet. The Al coating was uniform in thickness and had good bonding with the substrate during all the processing. This Al-coated Mg–Li alloy exhibited a good corrosion resistance in a 0.5 mass% HCl aqueous solution. No intermetallic compound was observed at the Al/Mg–Li interface after the extrusion and the rolling. The Al-coated Mg–Li alloy sheet exhibited an elongation to fracture of 35% at room temperature at a strain rate of 0.001 s^{-1} without any debonding between the coating and the substrate. When tensile tested at 573 K at 0.001 s^{-1} in the air, the Al coating remained undamaged even until an elongation of about 150%. Further elongation generated cracks on the coating and the specimen fractured at an elongation of about 200%. In an Ar atmosphere, the specimen exhibited a fracture elongation of over 400% under the same conditions at 573 K at 0.001 s^{-1} , although a large number of cracks generated on the Al coating.

Keywords Mg–Li alloy · Composite material · Coating · Mechanical properties · Superplasticity

1 Introduction

Light-weighting of engineering systems like vehicles and aircrafts can improve fuel efficiency. It is reported that 22.5 kg of mass reduction will improve fuel efficiency by around 1% in a typical car [1]. Therefore, development of lightweight structural materials is an important subject. Mg-based alloys with Li as a major alloying element, which are usually called Mg–Li alloys, have significant low densities ($1.35\text{--}1.65 \text{ g cm}^{-3}$) compared with other structural metals such as Al (2.70 g cm^{-3}) alloys and Fe (7.87 g cm^{-3}) alloys [2, 3]. Mg–Li alloy is the lightest structural metal in existing engineering alloy systems,

which has high specific strength and good castability. Furthermore, compared with conventional Mg alloys, the plasticity of Mg–Li alloys has a large improvement when the content of Li is higher than 5.5 wt%, because the stable phase changes from hcp ($< 5.5 \text{ wt\% Li}$) to hcp/bcc ($5.5\text{--}10.3 \text{ wt\% Li}$) and bcc ($> 10.3 \text{ wt\% Li}$) [4].

However, the largest disadvantage of Mg–Li alloys is their poor corrosion resistance. In addition, one of the most effective ways to prevent corrosion is coating. Conventional methods to produce coating are chemical methods, e.g., plating and anodizing [5–9]. However, Mg and its alloys are difficult to be coated by chemical methods for their active chemical properties [10]. To produce a good coating, complex procedures are usually needed, and sometimes, toxic solution is used.

Therefore, in this study, we attempted to produce a corrosion-resistant coating on Mg–Li alloys by a hot extrusion method instead of chemical ways. Mechanical ways like rolling and extrusion to produce protective coating on Mg alloys were proved to be effective, efficient, and environmental friendly by removing chemical treatments [11–13]. Al was chosen, as the coating material in this study for it exhibits a very good corrosion resistance in ambient environment and is also light metal. An Al-coated Mg–Li alloy is expected to exhibit a good corrosion

Available online at <http://link.springer.com/journal/40195>

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resistance without losing the advantage of light-weighting. Normally, these two metals cannot be bonded simply with each other because of the existence of oxide films on their surfaces. We consider that the oxide films can be broken by a severe plastic deformation, due to the enormous difference of ductility between metal and metal oxide. In addition, subsequently, fresh metal surfaces come to the front. The new metal surfaces are severely deformed in contact with each other under a very high pressure to avoid re-oxidation. Therefore, with the newly formed metal surfaces, the two metals can be strongly bonded. In addition, an Al-coated Mg–Li alloy bar was produced using the hot extrusion method in this study. After the extrusion, we rolled the Al-coated Mg–Li alloy bar into thin sheet, because sheet is more widely used to produce various shapes. In addition, for a further forming of the sheet, its mechanical properties, especially plasticity, are of great research value. In the previous study, though this kind of Al/Mg–Li alloy/Al clad sheet was produced with good corrosion resistance, the debonding between coating and substrate during further deformation was the problem [11].

Therefore, as stated before, the good plasticity of Mg–Li alloy is also an important reason for we choose it as the base alloy. Meanwhile, we noted that dual-phase Mg–Li alloys were reported a lot to exhibit superplasticity after severe deformation [14–16]. Owing to the large deformation of superplasticity, now, many complex parts can be processed by superplastic forming [17–19]. Moreover, a non-superplastic Al coating was reported to be superplastically formed with an AZ80 Mg alloy substrate without breaking [20]. From these facts, it is expected that an Al-coated Mg–Li alloy, which is our main focus, can also exhibit superplasticity with an unbroken Al coating. By superplastic forming, it is expected to process the Al-coated Mg–Li alloy sheet into a variety of desired shapes.

Thus, the aim of this study is to investigate (1) the feasibility of Al coating on an Mg–Li alloy by a hot extrusion process, (2) effects of the Al coating on the corrosion resistance, and (3) mechanical properties and superplasticity of the Al-coated Mg–Li alloy sheet.

2 Experimental

Figure 1 displays the schematic of the hot extrusion equipment. An Mg–Li alloy billet was put in the bottom of the container, and a 5 mm-thick Al plate (99.99 mass% purity) was placed between the die and the Mg–Li alloy billet. The Mg-8.03 mass% Li alloy was employed in this study. This composition was checked by an inductively coupled plasma atomic emission spectrometer (ICP-AES). The size and shape of the die are shown in Fig. 2. Before extrusion, the die and rams were pre-heated to 548 K

Fig. 1 Schematic of the hot extrusion equipment

Fig. 2 Schematic of the die

outside the container, while the container was pre-heated to 473 K. Then, the Mg–Li alloy billet and the Al plate were put into the container. In addition, the die and rams were set above the Al plate, as illustrated in Fig. 1. After this setting, the temperature of the container was increased from 473 to 523 K and the extrusion was carried out. The pressure was 300 kN and the extrusion speed was 0.6 mm/s. After extrusion, the as-extruded bar was cut every 10 mm in the longitudinal direction from the tip to the end to measure the thickness of the Al coating on the cross section. Five data measured on the long sides of rectangular cross section were chosen to calculate average.

The extruded bar was rolled from 8 to 1.5 mm in thickness at 523 K using a two-high mill. The rolling direction was parallel to the extrusion direction. After rolling, tensile specimens were cut from the as-rolled sheet using a wire electrical discharge machine. The dimensions of the tensile specimen are shown in Fig. 3. The tensile

Fig. 3 Schematic drawings of tensile test specimen: **a** top view, **b** longitudinal section

direction was parallel to the rolling and extrusion directions. Tensile tests were performed with the Instron tensile machine at room temperature at a strain rate of 0.001 s^{-1} in the air. High-temperature tensile tests were also performed at 573 K at a strain rate of 0.001 s^{-1} both in the air and in the an Ar atmosphere.

The microstructures of the as-extruded, as-rolled, and tensile-elongated samples were observed using an optical microscope (OM). To distinguish α phase and β phase in the Mg–Li alloy substrate, some samples were etched with a mixture of picric acid of 32 mL, acetic acid of 20 mL, distilled water of 20 mL, and ethyl alcohol of 18 mL before observation. The elements distribution and electron backscattering diffractions (EBSD) analyses were conducted by a JAMP-9500F Auger electron spectroscopy (AES). The fracture surfaces of the tensile specimens were observed by a JSM-6510LA scanning electron microscope (SEM).

The corrosion resistance was evaluated by an immersion test. Different samples were immersed in a 0.5 mass% HCl aqueous solution for 90 min at room temperature and their weight losses per unit area was monitored during the immersion. Before immersion, the surfaces of each sample except the Al-coating surface were covered with resin, which does not react with the acid. For a comparison, Mg–Li alloy without Al coating and the pure Al were also subjected to this corrosion test.

3 Results and Discussion

3.1 Feasibility of the Al-Coated Mg–Li Alloy Sheet

Figure 4a shows the cross section of the as-extruded Al-coated Mg–Li alloy. The Al-coating layer is uniformly

coated on the surface of the Mg–Li alloy substrate. Figure 4b illustrates the interface between Al-coating layer and Mg–Li alloy substrate. In the Mg–Li alloy substrate, the bright and the dark areas are α (hcp, Mg-rich) and β (bcc, Li-rich) phases, respectively. From the figure, no cracks are observed at the interface, which suggest a good bonding at the interface. Moreover, there are no obvious intermetallic compound (IMC) layers forming at the interface which is beneficial to mechanical properties, because IMCs are generally brittle and deteriorate the mechanical properties of the composite. Figure 5 demonstrates the change of the Al-coating thickness in the longitudinal direction of the as-extruded bar. The thickness of the coating near the tip is particularly thick, because at the start of the extrusion, the thick Al plate covers the top of the Mg–Li alloy billet. As the extrusion proceeds, the thickness of the coating becomes uniform and it was reduced to around 200 μm at a position about 50 mm away from the tip.

Figure 6 shows the microstructures of the as-rolled Al-coated Mg–Li alloy. The rolling direction corresponds to the traverse direction of the figure. The Al coating keeps uniform thickness in the rolling direction in Fig. 6a. The phases in the Mg–Li alloy substrate are elongated along the rolling direction, especially β phase (dark part in the Mg–Li alloy substrate in Fig. 6b), and are severely elongated, because it is much more ductile than α phase (bright part in the Mg–Li alloy substrate in Fig. 6b). Furthermore, cracks or IMC layers were not observed at the interface.

3.2 Room Temperature Tensile Properties of the Al-Coated Mg–Li Alloy Sheet

Figure 7 shows the stress–strain curves obtained from the tensile tests at room temperature. The as-rolled Al-coated Mg–Li alloy sheet exhibits an ultimate tensile strength of

Fig. 4 **a** Cross section of the as-extruded Al-coated Mg–Li alloy rectangular bar which is about 50 mm from the tip, **b** interface between Al coating and Mg–Li alloy substrate in the cross section. The extrusion direction corresponds to the normal to the figure

Fig. 5 Change of the coating thickness in the longitudinal direction of the as-extruded Al-coated Mg-Li alloy bar

about 160 MPa and a fracture elongation of about 35%. Figure 8 shows the longitudinal section of the fracture part of the tensile specimen, in which no cracks at the coating/substrate interface can be seen after the tensile elongation, which suggests that the deformation of the coating keeps good consistence with the deformation of the substrate. Finally, as more clearly shown in Fig. 8b, the coating exhibits a typical necking of a ductile material, while the substrate exhibits a slight degree of necking associated with a shear fracture.

And then, heat treatments were attempted to get a better plasticity. After a heat treatment at 573 K for 60 min, the fracture elongation can be improved to about 45% as the illustrated by the red curve in Fig. 8, accompanied by a marked drop on strength. On the other hand, the growth of IMC at the interface is an undesirable consequence after heating, as shown in Fig. 9a, and IMCs are usually brittle and have bad influence on mechanical properties. Figure 9b shows a line profile of the element distribution at the interface. Small platforms of the lines can be seen at the interface, suggesting the forming of the IMC. Notably, the

Fig. 7 Nominal stress-strain curves of the as-rolled Al-coated Mg-Li alloy sheet at room temperature at 0.001 s^{-1}

diffusion of Li does not stop at the interface like Mg and Al. It has diffused far into the Al coating. This will partly change the pure Al into an Al-Li alloy.

Figure 10 shows the fracture surfaces of the specimens after tensile tests at room temperature. In Fig. 10a, without heat treatment, the Al coating has a sharp edge after fracture because of necking. In addition, there is a smooth surface of the Al coating on the side close to the Mg-Li alloy. These are in agreement with the longitudinal section, as displayed in Fig. 8b. On the other hand, after a heat treatment at 573 K for 60 min, the fracture surface of the Al coating becomes rough with a two-layered structure, as shown in Fig. 10b. As illustrated in Fig. 9a, the inner layer should be the IMC layer. In addition, the deep gap between the substrate and the IMC layer suggests that the fracture may preferentially start from the IMC layer or the IMC/Mg-Li interface because of the low ductility of the IMC.

Fig. 6 **a** Longitudinal section of the as-rolled Al-coated Mg-Li alloy sheet, **b** interface between Al-coating and Mg-Li-alloy substrates in the longitudinal section. The rolling direction corresponds to the traverse direction of the figures

Fig. 8 Longitudinal section of the as-rolled Al-coated Mg–Li alloy sheet elongated to fracture at room temperature: **a** low magnified image, **b** high magnified image of the Al-coating part

Fig. 9 **a** SEM image of the intermetallic compound layer, **b** AES line profile showing the distribution of Li, Mg and Al at the Al/Mg–Li alloy interface after a heat treatment at 573 K for 60 min

Fig. 10 SEM images of the tensile fracture surface of the specimen: **a** as-rolled specimen without heat treatment, **b** specimen after a heat treatment at 573 K for 60 min

3.3 Superplasticity of the Al-Coated Mg–Li Alloy Sheet

Figure 11 illustrates the EBSD grain and phase image of the as-rolled Al-coated Mg–Li alloy specimen just before the superplastic tensile test starting at 573 K, which is in fact a specimen after a heat treatment at 573 K for 10 min according to the experimental procedures. In the grain map in Fig. 11a, in the Mg–Li alloy substrate, grains of both α and β phases are elongated along the rolling direction: the width of the elongated grain in the normal direction is less than 10 μm , while the lengths of many grains in the rolling direction are larger than 20 μm . The grains of Al are equiaxed and are most less than 10 μm in size.

Figure 12a shows the stress–strain curves of the specimens tensile tested at different temperatures from 448 to 573 K at 0.001 s^{-1} . At above 473 K, the specimens can exhibit fracture elongations of about 200%, while at 448 K, the fracture elongation decreases to about 170%. In addition, as testing temperature rising, the flow stress falls dramatically. On the other hand, Fig. 12b shows the stress–strain curves at the same temperatures at the strain rate of 0.01 s^{-1} . Compared with Fig. 12a, flow stresses are larger at the same temperature in Fig. 12b, while the fracture elongation decreases by about 50% at every temperature.

In Fig. 12a, oscillations can be observed on all the curves, which means that hardening stage and softening stage alternatively occur during the tensile process. Dynamic recrystallization (DRX) is an important softening mechanism during the hot deformation of Mg–Li alloy [21]. Comparing Fig. 12a, b, the softening stages are obvious in (a) at lower strain rate, while no large rise and fall can be observed in (b) at rather high strain rate. This is because that DRX is a process induced by atomic diffusion [22]: at lower strain rate in Fig. 12a, it takes relatively long

time to reach the same strain, and then, there is enough time for diffusion to induce the DRX, and in Fig. 12b, at very high strain rate, there is little time for diffusion; therefore, we cannot see an obvious softening stage caused by DRX.

The superplastic deformation can be described by the model as follows [23]:

$$\sigma = k\dot{\epsilon}^m,$$

where σ is the flow stress, $\dot{\epsilon}$ is the strain rate, constant k depends on composition, microstructure, and temperature, and m is the index of strain rate sensitivity. The m value is important in describing the behavior of superplastic deformation. In addition, a large m value usually means a large superplastic elongation. A typical superplasticity is characterized by an m value of more than 0.3 [24]. In addition, according to the above equation, the m value can be calculated by the slope of a double logarithmic plot of flow stress versus strain rate:

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}}.$$

Therefore, the m values of the Al-coated Mg–Li alloy sheet at different temperatures are calculated shown in Fig. 13. It is obvious that the m value increases as the temperature rising. In addition, the largest value is 0.38 at 573 K. Notably, the value of m falls greatly from 0.32 to 0.25 when temperature decreases from 473 to 448 K. This is in consonance with the tensile results in Fig. 12, where obvious fall of elongation can be seen when temperature drops to 448 K.

On the other hand, when temperature is higher than 473 K, the fracture elongations are almost the same, though the m value changes. In fact, we found that cracks always occurred on the Al coating after the specimen was elongated to about 150% no matter the temperature.

Fig. 11 EBSD image of the specimen before superplastic tensile test: **a** grain map, **b** phase map

Fig. 12 Stress–strain curves of the specimens tensile tested at temperatures from 448 K to 573 K in the air at strain rates of **a** 0.001 s^{-1} , **b** 0.01 s^{-1}

Fig. 13 Effects of strain rate on the flow stress of the Al-coated Mg–Li alloy sheet at different temperatures

Figure 14 shows the longitudinal section of the specimen after an elongation of 150% at 573 K at 0.001 s^{-1} . Though the Al coating is unbroken, some small necking parts can be seen. When the tensile test continued to larger than 150% elongation, cracks started to generate on the Al coating, and consequently, the specimen fractured at an elongation of about 200% in the air, as shown in Fig. 15. On the other hand, when tested in an Ar atmosphere, the fracture elongation increases to more than 400%. If observed carefully, there are many cracks on the Al coating of the specimen deformed in an Ar atmosphere. In contrast, the specimen deformed in the air has a small number of cracks only near the fracture part. The oxidation of the Mg–Li alloy during superplastic deformation in high-temperature air has been reported in some researches [25, 26]. In the case of this study, after a crack appears on the Al coating, the Mg–Li alloy substrate becomes exposed to the

Fig. 14 Longitudinal section of tensile specimen of the as-rolled Al-coated Mg–Li alloy sheet after a tensile elongation of 150% at 573 K at 0.001 s^{-1} in the air

outside atmosphere. In the air, oxidation takes place and it should weaken the substrate, where the crack generates and then cause a strain concentration there. Finally, a rapid fracture will occur after the crack appears. Differently, in an Ar atmosphere, the Mg–Li alloy substrate will not suffer oxidation even if the crack appears on the Al coating, which mean that there is no strain concentration at the crack. Therefore, the specimen exhibits much larger elongation with many cracks on the Al coating.

These cracks on the Al coating should cause low corrosion resistance. In the previous study of our group, we investigated the elongation of an Al-coated AZ80 Mg alloy, which does not contain Li element. No crack was observed on the Al-coating surface even after a very large elongation of 550% at 573 K at 0.001 s^{-1} [20]. Therefore, the Li element is considered to be an important factor in the

Fig. 15 Tensile specimens of the Al-coated Mg-Li alloy at 573 K at 0.001 s^{-1} in the air and in an Ar atmosphere

Fig. 16 Cracks on the Al coating along about 45° to the tensile direction

crack formation in the present case. As shown in Fig. 9b, Li can diffuse far into the Al coating at high temperature. From these results, it can be considered that because of the high diffusivity of the Li atom, the pure Al became an Al-Li alloy with a small content of Li. Al-Li alloys are known to have anisotropic mechanical properties after rolling and it exhibits low yield strength in the directions of 45° – 60° to the rolling direction [27]. In this study, crack formation in about 45° to the tensile as well as rolling direction was observed on the Al-coating surface, as shown in Fig. 16. Therefore, cracks on the Al coating may be related to the diffusion of Li into the Al-coating layer from the Mg-Li alloy substrate.

Fig. 17 Weight loss of different samples in 0.5 mass% HCl aqueous solution

3.4 Corrosion Resistance of the Al-Coated Mg-Li Alloy Sheet

Figure 17 displays the weight losses of some different alloys during immersion in 0.5 mass% HCl aqueous solution at room temperature. Without the Al coating, as marked by the circle, the Mg-Li alloy reacts with the acid solution seriously and loses its weight very quickly. However, both the as-extruded and as-rolled Al-coated Mg-Li alloy samples have no weight loss in the HCl solution, the same as the result of pure Al. Therefore, it has been demonstrated that an excellent corrosion resistance can be given to the Mg-Li alloy by Al coating using the present hot extrusion method and that the further rolling process causes no adverse effect on the corrosion resistance of the Al-coated Mg-Li alloy. Moreover, the as-rolled Al-coated Mg-Li alloy specimen elongated to 150% at 573 K at 0.001 s^{-1} in the air also exhibits the same good corrosion resistance as the pure Al. This requires the coating to exhibit a same elongation of 150% as the substrate to keep unbroken. In addition, this excellent ductility of the Al coating is revealed in the following investigation of the tensile properties.

4 Conclusions

1. An Al-coated Mg-8 mass% Li alloy rectangular bar was successfully fabricated by hot extrusion. A uniform and well bonded Al coating was produced with a thickness of approximately $200 \mu\text{m}$. After rolled into thin sheet, the coating kept uniform thickness and the bonding between coating and substrate remained free from cracks.

2. Tensile test of the as-rolled Al-coated Mg–8Li alloy sheet exhibited a fracture elongation of about 35% at room temperature at 0.001 s^{-1} . There is no break at the bonding between the substrate and coating.
3. The as-rolled Al-coated Mg–Li alloy sheet exhibited superplastic elongations of about 200% and 400% at 573 K at 0.001 s^{-1} in the air and in an Ar atmosphere, respectively.
4. The Al-coated Mg–Li alloy sheet exhibited excellent corrosion resistance in a 0.5 mass% HCl aqueous solution. Even though after a large elongation of 150%, this good corrosion was still kept.

Acknowledgements This work was partly supported by the Nanotechnology Platform Program (Molecule and Material Synthesis) of the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

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