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*Research Article*

# Effect of Prior Rolling on Microstructures and Property of Diffusion-Bonded Mg/Al Alloy

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In this study, magnesium alloy AZ91, which was cast by double roll casting system, was rolled by a rolling mill. Then, rolled magnesium alloy and magnesium alloy without being rolled were, respectively, welded with aluminum alloy 6061 by diffusion bonding method. Furthermore, annealing process was applied to refine the microstructure and improve mechanical property. The microstructure and elemental distribution of diffusion zone were investigated with a scanning electron microscope (SEM), an electron probe micro analyzer (EPMA), and a transmission electron microscope (TEM). In addition, hardness and tensile strength were measured. When cast magnesium alloy was used, the width of diffusion layers was wider than that with rolled magnesium alloy. And the width increased with the increasing annealing temperatures. Element distribution of specimens with annealing was more uniform than that did not undergo annealing process. Furthermore, tensile strength turns to be strongest after annealing at 250°C. And the strength of the specimens with rolled magnesium alloy was stronger than that with cast magnesium alloy which was not rolled.

## 1. Introduction

With the rapid development of the transportation, aerospace, national defense, and military industry, magnesium alloys and aluminum alloys are both widely used in aerospace, automotive, machinery, and electrical and chemical industry [1–4]. In addition, with growing energy, economy, and environmental needs, Mg alloys have become the favorite choices in automotive industry [5]. If aluminum alloy can be bonded with magnesium alloy and form a kind of composite material, not only would the flexibility and availability be improved substantially but also the weight and cost would be reduced obviously. So it is significant to achieve reliable connection of Mg/Al dissimilar metals. Presently, there are many researchers studying in this field.

Fusion welding is one of the most widely used methods for the joining of metals [6]. And there are many other welding methods which have been used to join Mg alloys and Al alloys such as soldering, electron beam welding [7], resistance spot welding, explosive welding [8, 9], laser welding, and vacuum diffusion bonding applied in this study [10, 11]. However, no matter which technique is used, the brittle and hard intermetallic compounds such as $Al_3Mg_2$ and $Mg_{17}Al_{12}$ were formed in the joints as annealing can transform the form of crystal and improve the defect of microstructures and rolling process can refine the microstructures. Therefore, brittleness is reduced and mechanical characters turn to be better. In this study, in order to improve the microstructures and mechanical properties, rolling process on magnesium alloy before diffusion bonding was carried out, and the welded specimens were annealed at different temperatures. In addition, microstructures and properties were investigated. In diffusion process of this paper, specimens used for diffusion bonding were put into the device justly and cannot move along the longitudinal direction. No pressure is added to the specimen before heating. But pressure will generate due to thermal expansion. And it is nearly the first time the different effect of cast magnesium alloy

Table 1: The parameters of rolling process.

| Pass number | Thickness (mm) | Temperature (°C) | Reduction (%) | Rolling speed (m/min) |
|---|---|---|---|---|
| 0 | 2 | 250 | | |
| 1 | 1.4 | 250 | 30 | 2 |
| 2 | 1.0 | 250 | 29 | 2 |

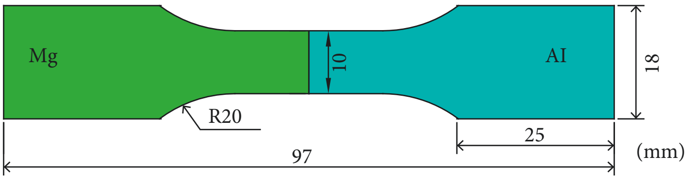


Figure 1: Dimensions of specimens for diffusion bonding.

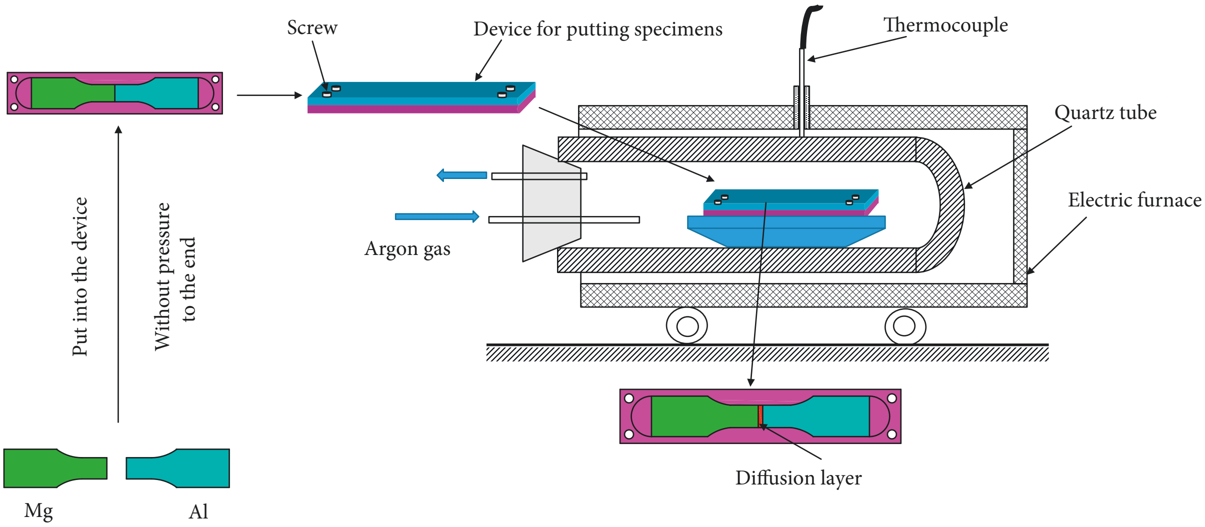
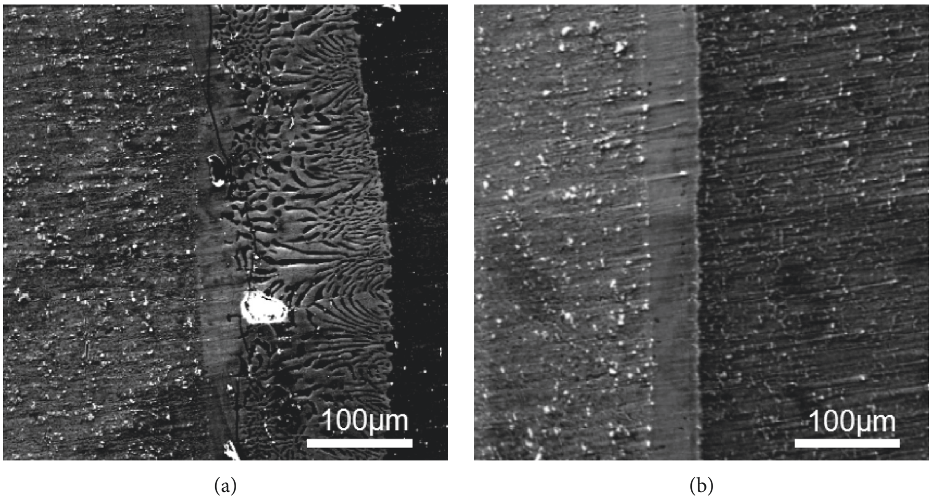


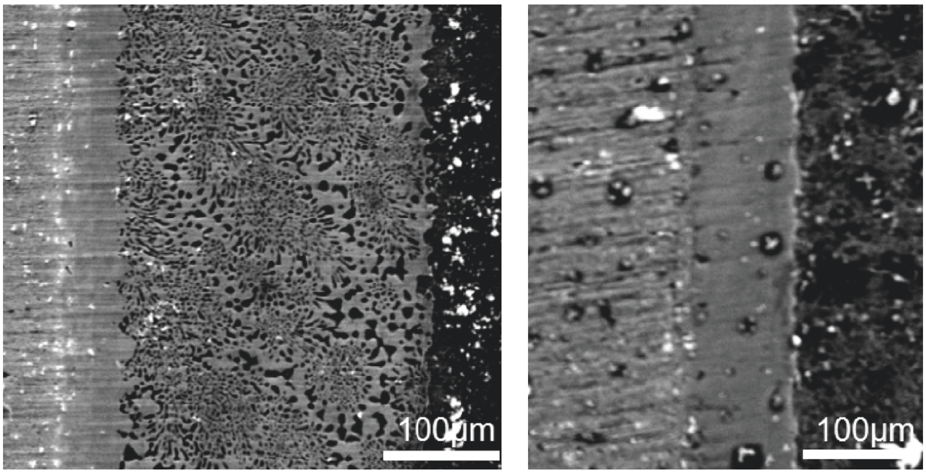
Figure 2: Diagrammatic sketch of diffusion bonding process.



(a)

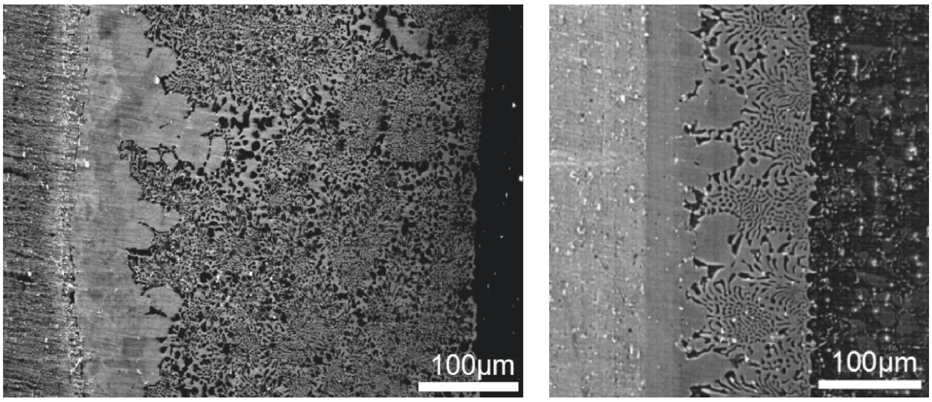
(b)

Figure 3: SEM micrograph of joints annealed at 200°C: (a) without rolling; (b) with rolling.



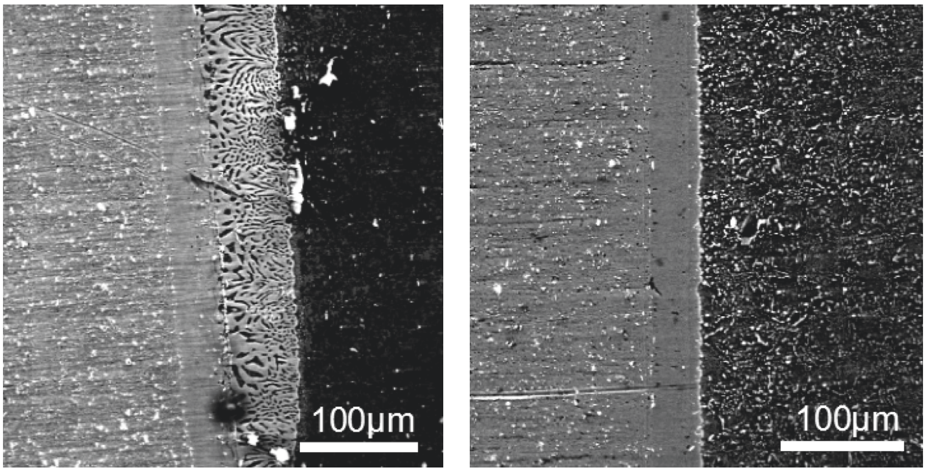
(a) (b)

Figure 4: SEM micrograph of joints annealed at 250°C: (a) without rolling; (b) with rolling.



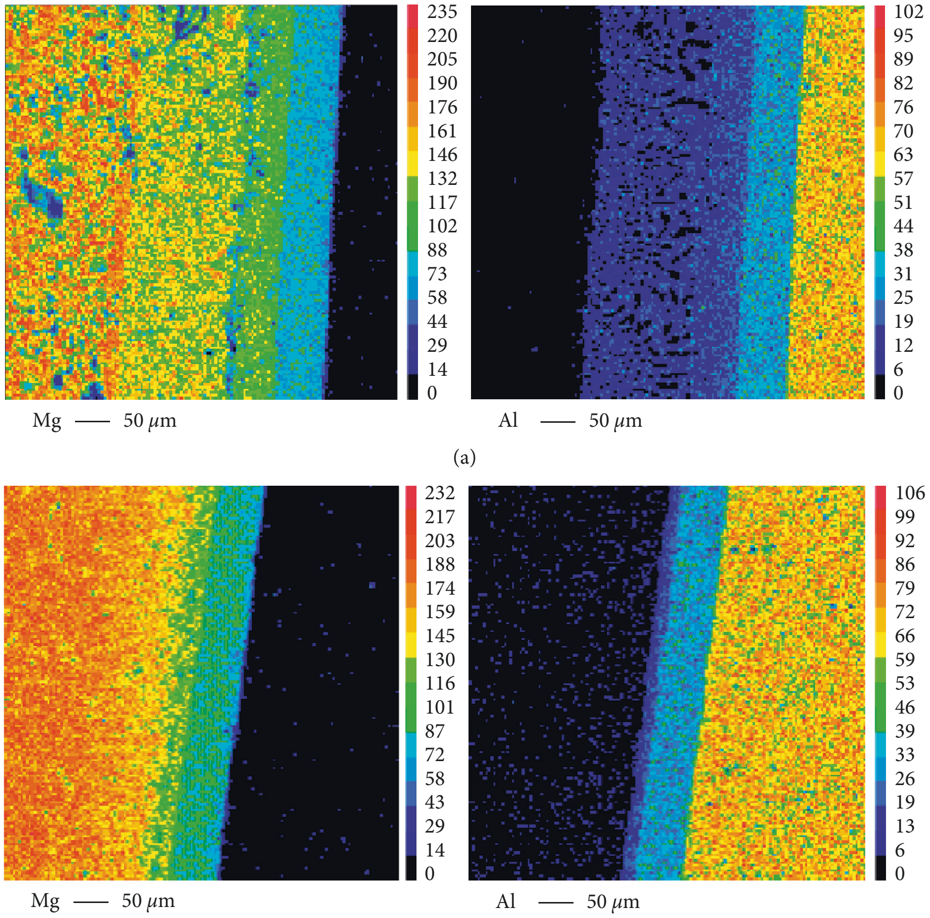
(a) (b)

Figure 5: SEM micrograph of joints annealed at 300°C: (a) without rolling; (b) with rolling.



(a) (b)

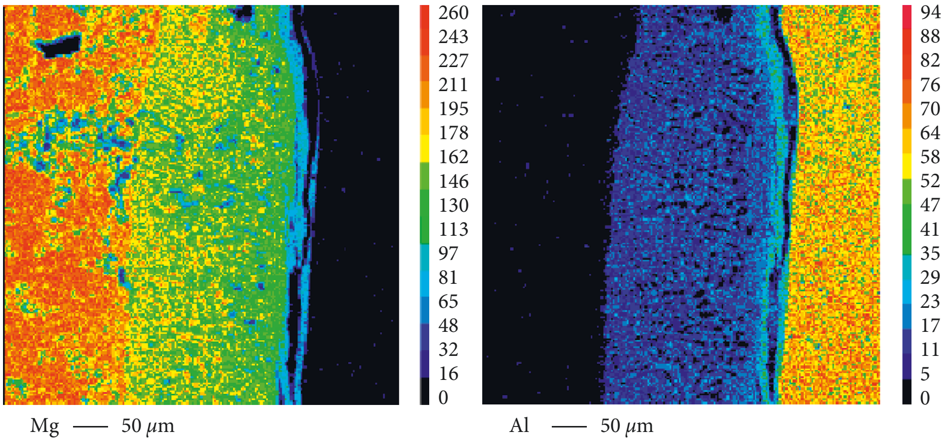
Figure 6: SEM micrograph of joints without annealing: (a) without rolling; (b) with rolling.



(a)

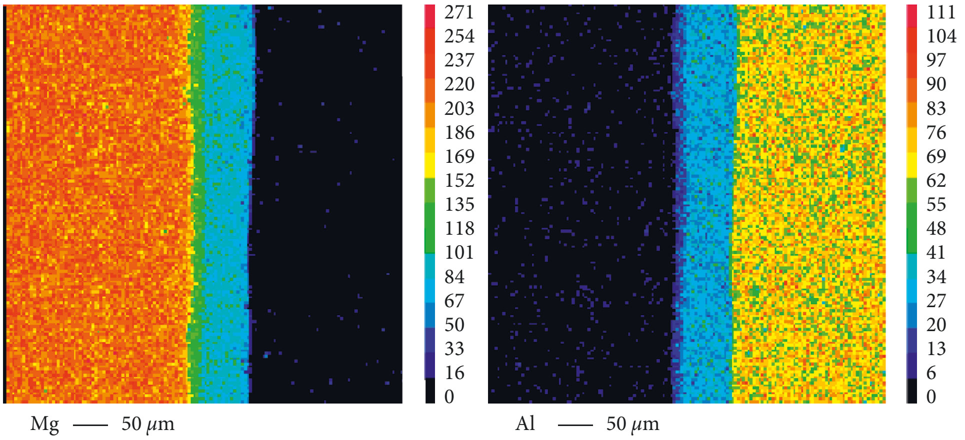
(b)

Figure 7: Elemental distribution without annealing: (a) without rolling; (b) with rolling.



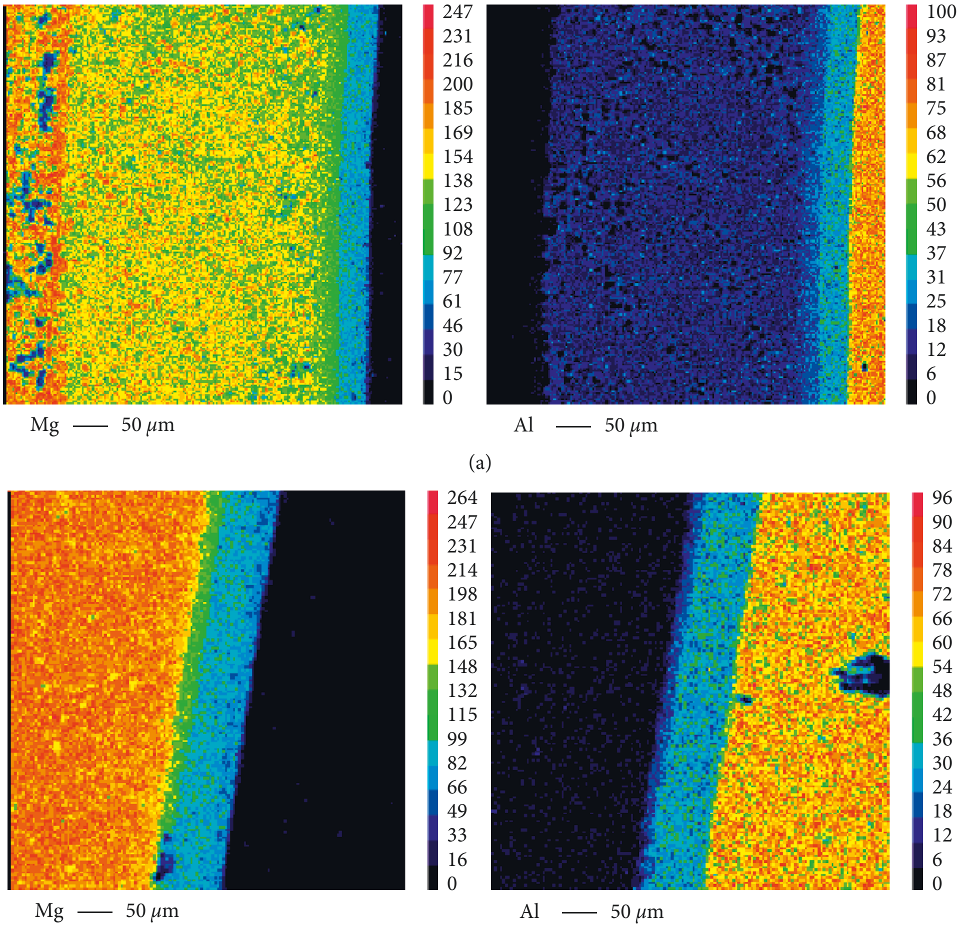
(a)

Figure 8: Continued.



(b)

Figure 8: Elemental distribution annealed at 200°C: (a) without rolling; (b) with rolling.



(a)

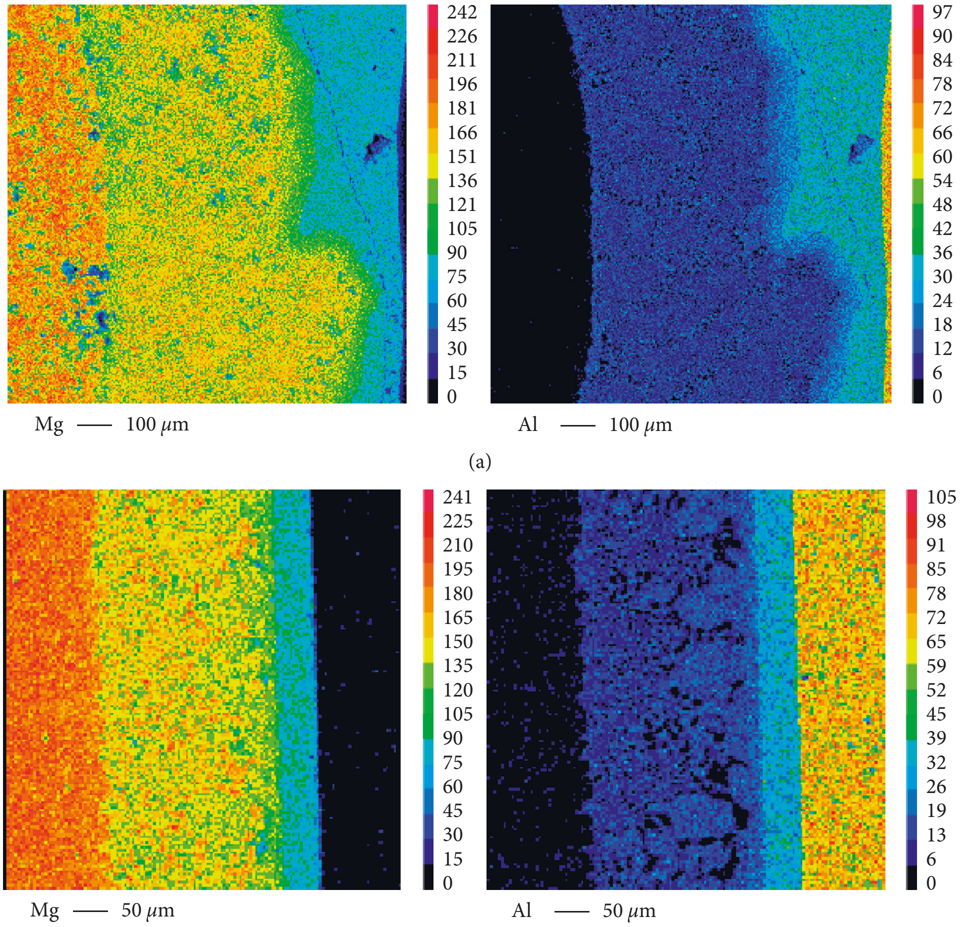
(b)

Figure 9: Elemental distribution annealed at 250°C: (a) without rolling; (b) with rolling.

and rolled magnesium alloy on diffusion bonding between magnesium alloy and aluminum alloy using this process was studied. Based on the results of this study, the application of diffusion bonding and composite material of magnesium alloy and aluminum alloy will be used widely.

## 2. Materials and Methods

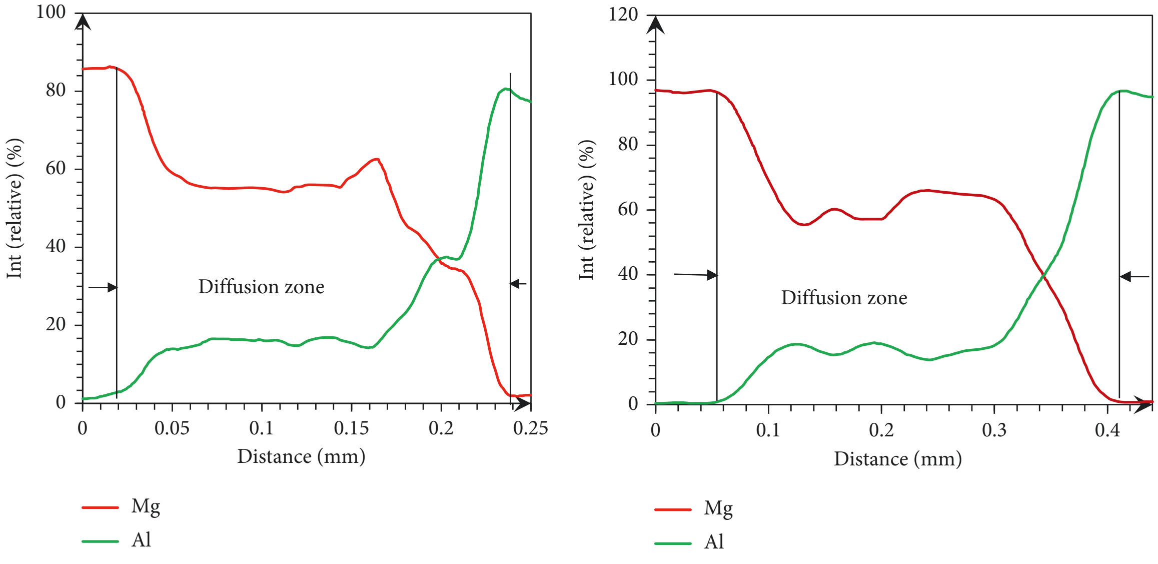
In this study, the AZ91 magnesium alloy and 6061 aluminum alloy were used for diffusion bonding and annealing process. Experiments were also carried out via using SEM, XRD,



(a)

(b)

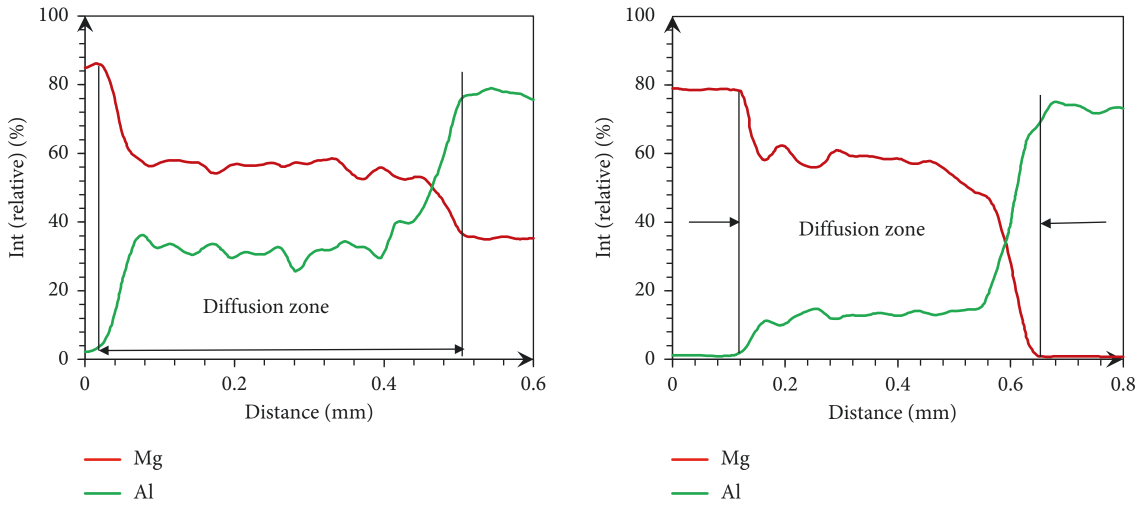
Figure 10: Elemental distribution annealed at 300°C: (a) without rolling; (b) with rolling.



(a)

(b)

Figure 11: Continued.



(c)

(d)

FIGURE 11: Result of line scanning for elemental distribution, with cast magnesium alloy: (a) without annealing and annealing at (b) 200°C, (c) 250°C, and (d) 300°C.

TEM, Vickers hardness tester, and tensile machine to evaluate the microstructure, crystal structure, and mechanical behavior.

*2.1. Experimental.* As the first step in the experimental procedure, a 6-high rolling mill with the rolls diameter of 80 mm was used to roll AZ91 magnesium alloy sheets from 2 mm to 1 mm. The parameters of rolling process are shown in Table 1.

The second step in the experimental procedure was to cut AZ91 magnesium alloy sheets and 6061 aluminum alloy sheets to the dimensions shown in Figure 1.

Then, the oxide layers on the surface of substrate were polished with abrasive papers and the ground samples were wiped with acetone and then put into the device justly. Samples cannot move along longitudinal direction without pressure to the ends. The device was put into the electric furnace, joining temperature was set at 440°C according to the Mg-Al phase diagram, and holding time was 60 min; after cooled down to room temperature in electric furnace, specimens were successfully joined in the equipment with the atmosphere of argon. The flow chart of diffusion bonding is shown in Figure 2.

Besides, in order to improve the microstructures and mechanical properties, the welded samples were used for the annealing treatment experiments. According to the Mg-Al phase graph and previous annealing experience, the samples were annealed using heat treatment temperatures of 200, 250, and 300°C, and the holding time was 60 min. After heat treatment, samples were cooled to room temperature in an electric furnace.

For the purpose of studying the effect of rolling process and annealing temperatures on microstructures and the properties of the interfaces, a series of specimens annealed at different conditions were cut across the diffusion zone. The cut sections were then inlaid into resin to facilitate the investigation of microstructures. Using a grinder and abrasive papers (GRIT 240, 600, 800, and 1200), the samples were ground and polished with a polishing compound. The microstructures and elemental distribution of the joints were studied, respectively, by SEM, XRD, TEM, and EPMA. Then, tensile strength and Vickers hardness were investigated.

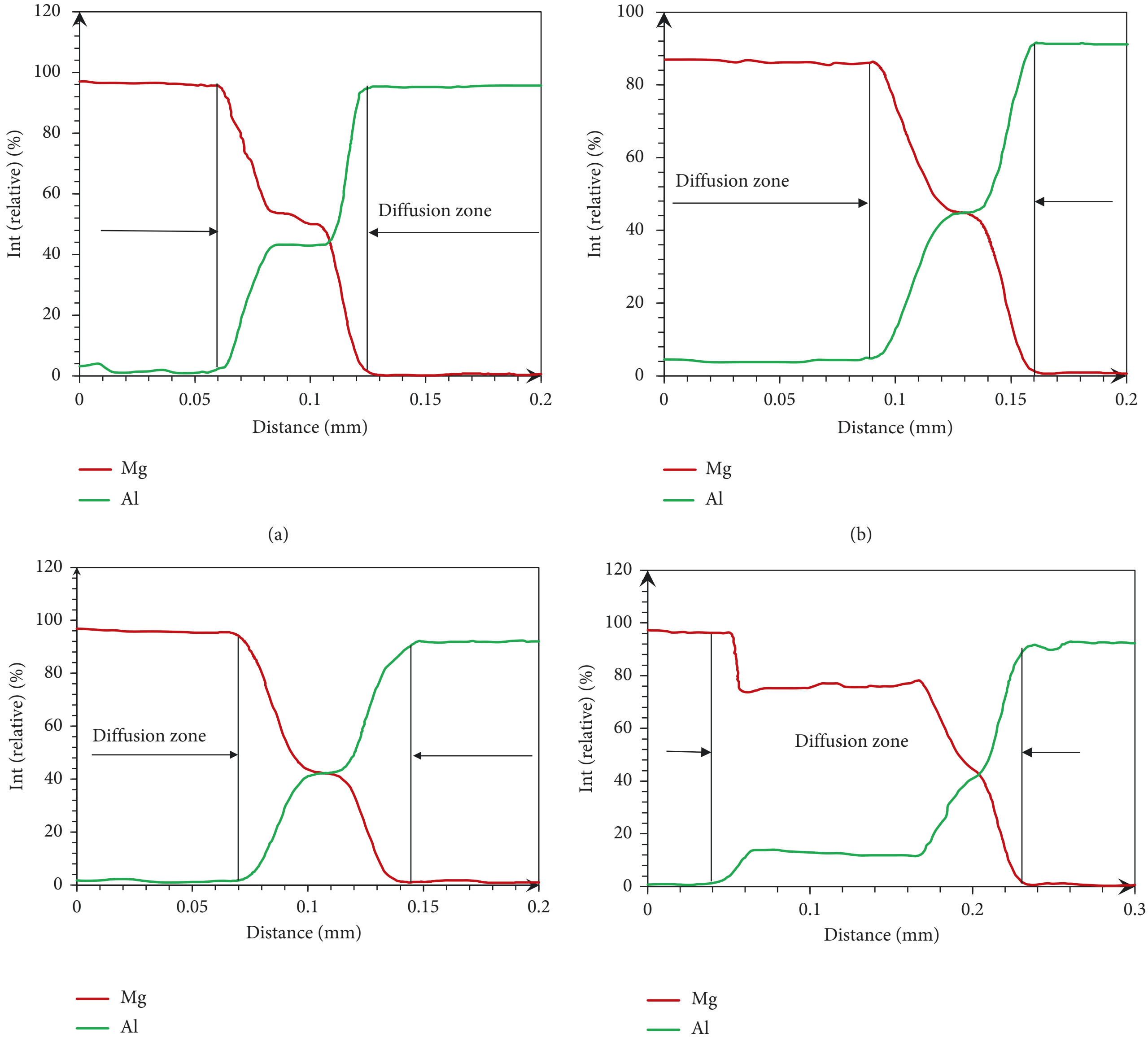
## 3. Results and Discussions

*3.1. Microstructures of Joints.* The microstructures of the bonding interfaces annealed at 200°C, 250°C, and 300°C were therefore observed. The microstructures of the joints are shown in Figures 3–5. Figure 6 shows the microstructures of joints that did not undergo annealing.

Based on the SEM micrographs above, the conclusions can be obtained that as the annealing temperature rises, the width of the diffusion layers increases. This is because the diffusion rate increases with increasing temperature. Moreover, interface of the specimens with rolled magnesium alloy is thinner than that with cast magnesium alloy. The reason is that rolling process makes the atomic spacing of magnesium alloy decrease, and then it is difficult for atoms to diffuse between magnesium alloy and aluminum alloy, so the width of diffusion layers becomes thinner.

The results of surface scanning for elemental distribution are shown from Figures 7–10, and the same conclusions with SEM micrographs can be obtained. In addition, when rolling process was applied, the element concentration of joints turns to be more than without rolling because the atom spacing decreases, so the atomic density increases.

Figures 11 and 12 are the results of line scanning for elemental distribution. When cast magnesium alloy was used, corresponding to the conditions of no annealing, annealed at 200°C, 250°C, and 300°C, the thicknesses of diffusion layers were, respectively, 0.22 mm, 0.35 mm,



(a) (b) (c) (d)

Figure 12: Result of line scanning for elemental distribution, with rolled magnesium alloy: (a) without annealing and annealing at (b) 200°C, (c) 250°C, and (d) 300°C.

0.48 mm, and 0.51 mm. However, in case of rolled magnesium alloy, they exhibited thicknesses of 0.065 mm, 0.07 mm, 0.075 mm, and 0.19 mm. So, the same conclusions with SEM micrographs and surface scanning for elemental distribution can be obtained.

For the purpose of analyzing and identifying the crystal structure, investigation of welded specimens using XRD was carried out. At the same time, AZ91Mg alloy and 6061Al alloy were also investigated. The results are shown in the following figures. Figure 13 shows the diffraction diagram of diffusion layers. It can be depicted that the diffraction peaks of Mg side, Al side, and diffusion layers were different from each other. The diffraction peaks of diffusion layer on Mg side occurred at the place of $2\theta$ 67.3° and 72.5°. In case of Al side, the locations of peaks were 37.9°, 44.2°, and 81°. The peaks of diffusion layer near Mg were at the place of $2\theta$ 77.5°, 77.8°, and 77.9°, while diffusion layer near Al exhibited the peaks' locations of 37.9°, 44.2°, and 81°.

Figures 14 and 15 are the diffraction results of AZ91Mg and 6061Al alloy. If Figure 13 was compared with Figures 14 and 15, the conclusions can be obtained that the planes of Al in diffusion layers were (111), (200), and (222), while the planes of Mg in diffusion layers were (200) and (004).

For the purpose of identifying crystal structure of intermetallic compounds in diffusion zone further, experiments with the usage of TEM were carried out. And the results are shown in Figures 16 and 17.

According to the scale in Figure 16 and the measurement of the distance to the center, the actual interplanar spacing was calculated; after compared with the database, the

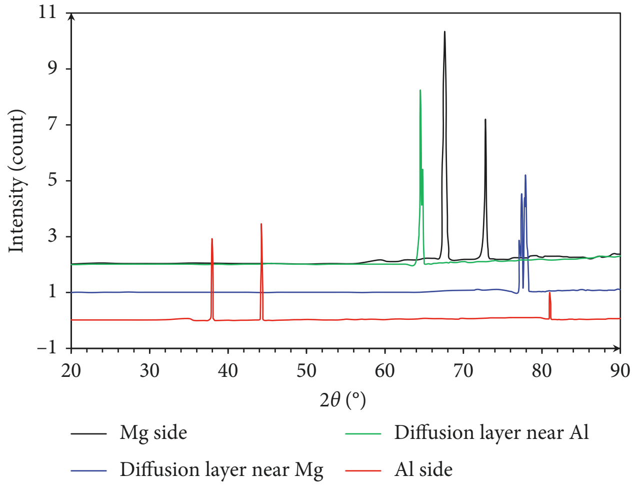


Figure 13: Diffraction diagram of diffusion layers.

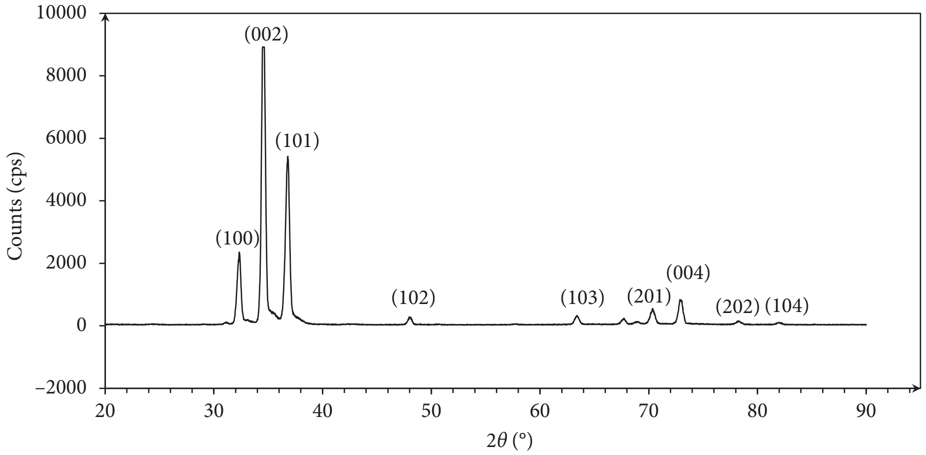


Figure 14: Diffraction diagram of AZ91Mg alloy for identification.

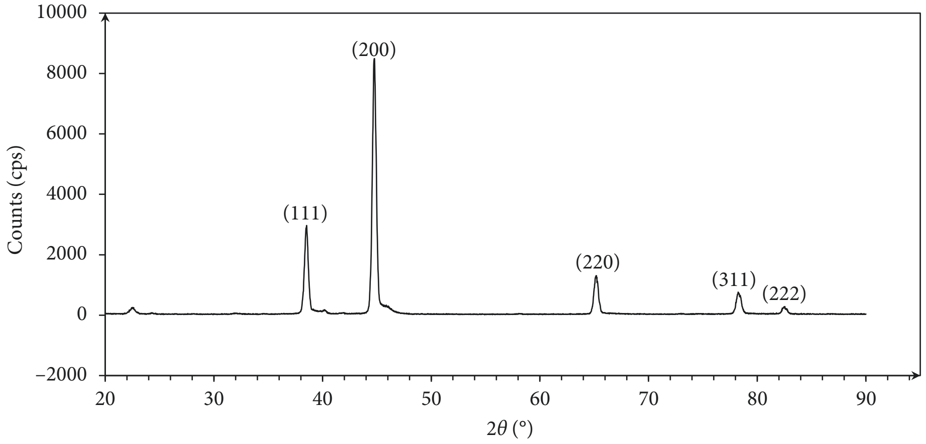


Figure 15: Investigation result of 6061Al alloy for identification.

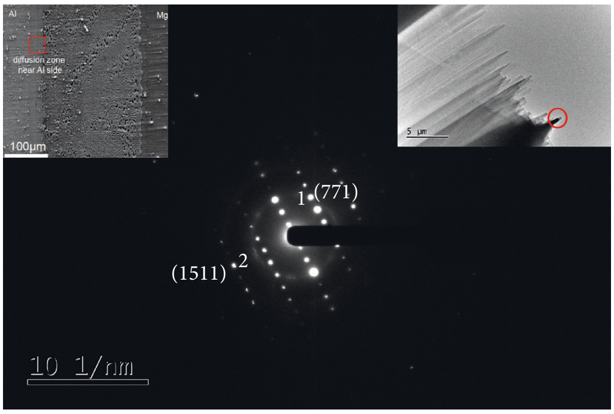


Figure 16: TEM micrograph of joints near Al side.

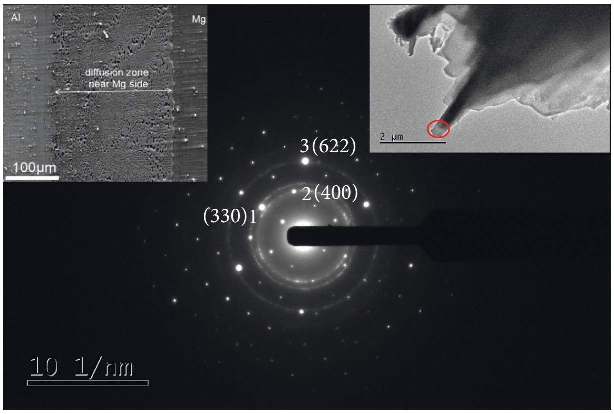
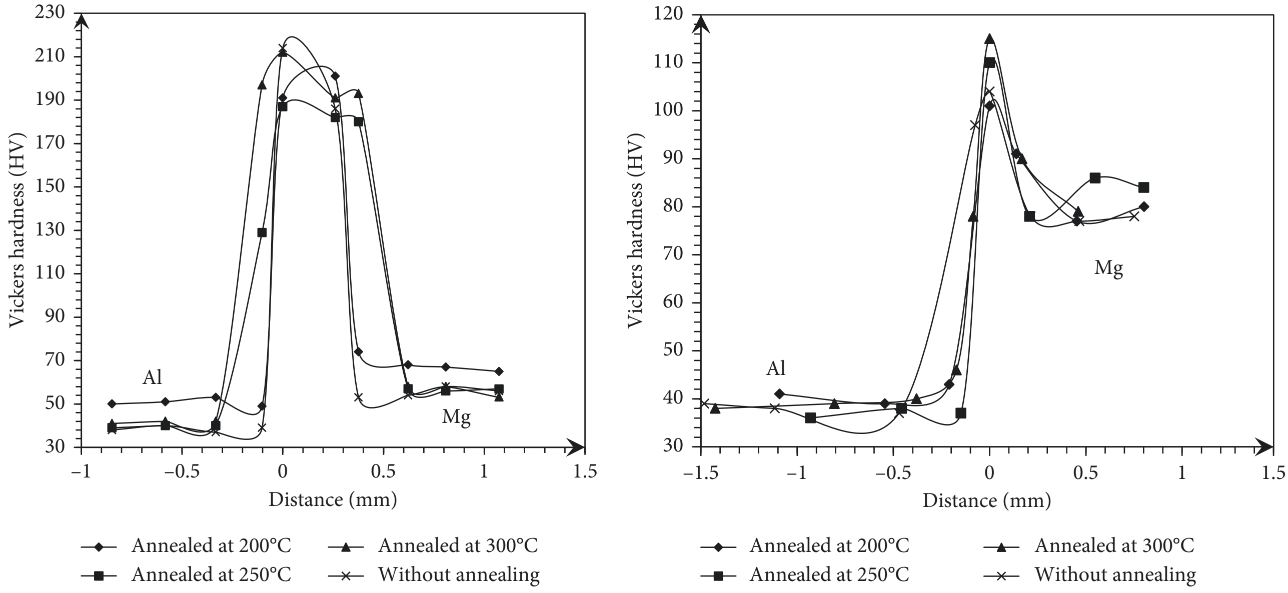


Figure 17: TEM micrograph of joints near Mg side.



(a) (b)

Figure 18: Hardness of diffusion zone: (a) with cast magnesium alloy; (b) with rolled magnesium alloy.

Figure 19: The shape and dimensions of the welded samples.

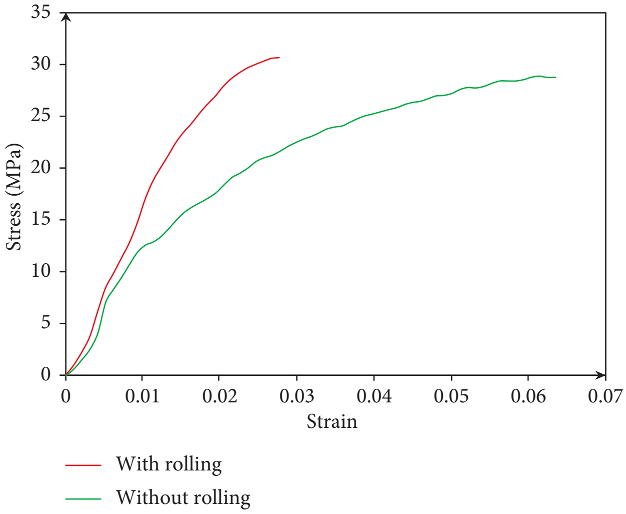


Figure 20: Stress-strain curve, without annealing.

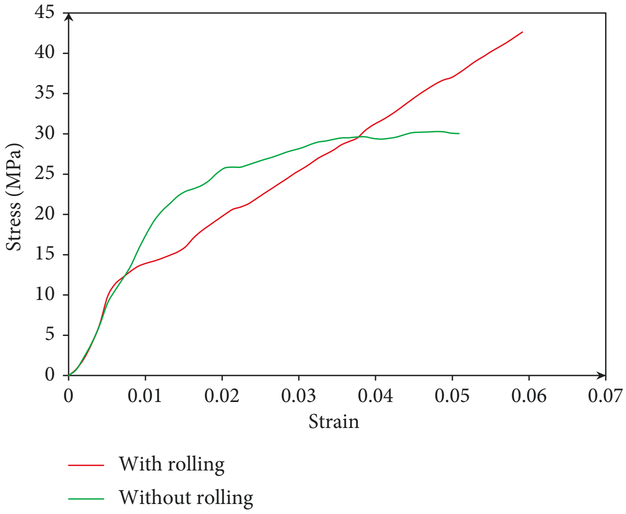


Figure 21: Stress-strain curve, annealing at 200°C.

conclusion was obtained that $d_1$ is 0.283 nm, $d_2$ is 0.187 nm. The results indicated that the crystal structure of the (771) and (1511) planes of $Al_3Mg_2$ is face-centered cubic.

Based on the scale in Figure 17 and the distance to the center, the interplanar spacing can be calculated. Therefore, the plane index can be confirmed by referring to the database. The interplanar spacings $d_1$, $d_2$, and $d_3$ are, respectively, 0.2480 nm, 0.2640 nm, 0.1600 nm. So, the corresponding plane index is (330), (400), and (622) of $Al_{12}Mg_{17}$ whose crystal structure is body-centered cubic.

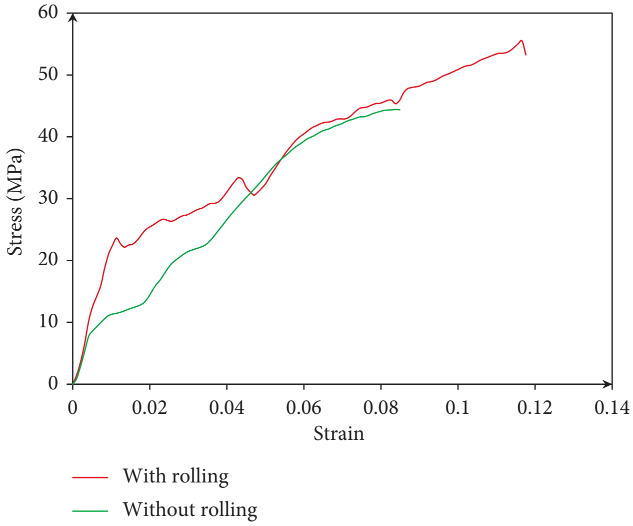


Figure 22: Stress-strain curve, annealing at 250°C.

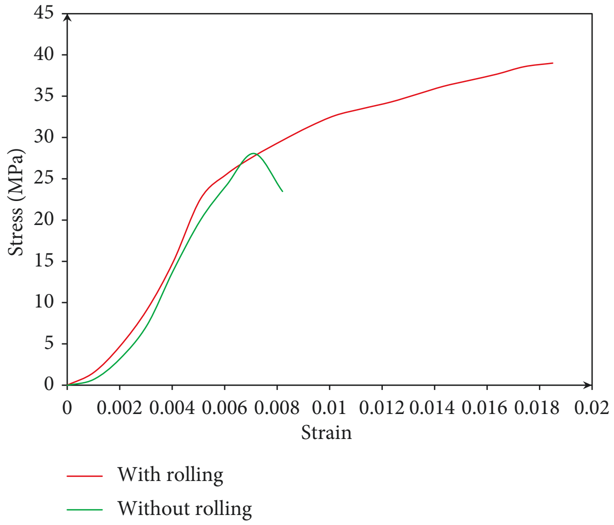


Figure 23: Stress-strain curve, annealing at 300°C.

For studying the effect of prior rolled magnesium alloy and annealing on mechanical property of diffusion-bonded Mg/Al alloy, microhardness of diffusion layers annealed at different temperatures was investigated, with the load set to be 1 kg. The distributions of hardness in the annealed samples are shown in Figures 18(a) and 18(b). The trends in the hardness distributions are broadly similar in that the hardness of the Mg side is higher than that of the Al side and significantly increases in the diffusion zone. When cast magnesium alloy was used, in the condition of no annealing and annealing at 200°C, 250°C, and 300°C, the highest hardness in the diffusion bonding region was, respectively, 214 HV, 201 HV, 187 HV, and 212 HV, which is shown in Figure 18(a). However, in the case of rolled magnesium alloy, the samples exhibited the highest hardness in diffusion zone of 115 HV, 110 HV, 101 HV, and 104 HV in Figure 18(b).

The shape and dimensions of the samples using for the test of tensile strength are shown in Figure 19.

The results are shown from Figures 20–23. The conclusions can be obtained that tensile strength increases after annealing treatment; when with cast magnesium alloy, the maximum of strength is 44 MPa, which was obtained after being annealed at 250°C. As what has been reported in other study, the strength is about 37 MPa when without annealing [12]. However, when with rolled magnesium alloy, the maximum of strength increases to 53 MPa, which was also achieved after being annealed at 250°C. So, it can be thought that the most appropriate annealing temperature is 250°C [13]. Furthermore, rolling and annealing process can refine microstructure, and the mechanical character can be improved.

It is said that a fine-grained material is stronger than one that is coarsely grained because it has a greater total grain boundary area to impede dislocation motion. For many materials, the yield strength, $\sigma_s$ varies with grain size according to the following equation:

$$\sigma_s = \sigma_0 + k \cdot d^{-1/2}. \tag{1}$$

In this expression, $d$ is the average grain diameter and $\sigma_0$ and $k$ are constants for a specific material [14]. According to this equation, it can be known that for one metallic material, the smaller the average grain diameter is, the stronger the yield strength will be. So, it is significant to use any processing to refine grain structure of metallic materials, in order to enhance its strength. In this study, as annealing and rolling can refine microstructure and make the grain diameter decrease, tensile strength increases.

## 5. Conclusions

Based on the results above, it can be thought that rolling process on magnesium alloy before welding and annealing after welding can refine the microstructure and have a good effect on tensile strength. The conclusions can be summarized as follows:

(1) For the specimens with rolled magnesium alloy, the width of diffusion zone is thinner than the specimens which were made with the magnesium alloy without rolling. Rolling process can refine microstructure, so the microstructures and elements distribution are more uniform.

(2) Intermetallic compound in diffusion zone near Al alloy is $Al_3Mg_2$ whose crystal structure is face-centered cubic, while the one near Mg side is $Al_{12}Mg_{17}$ whose crystal structure is body-centered cubic.

(3) Annealing can refine microstructure and improve mechanical property. After being annealed at 250°C, the strength turns to be the strongest. Furthermore, the strength of the specimen which was with rolled magnesium alloy is stronger than the one with cast magnesium alloy that was not rolled.

(4) The width of diffusion layers turn to be wider with the increasing annealing temperatures, and 250°C is the most appropriate annealing temperature.

## Data Availability

No data were used to support this study.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

## Acknowledgments

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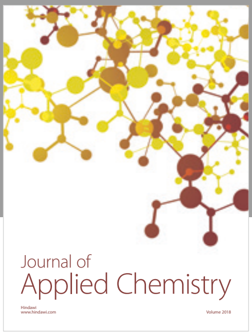
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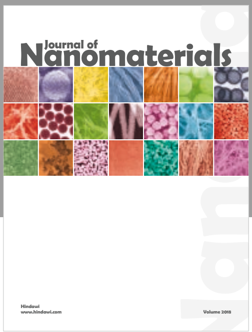
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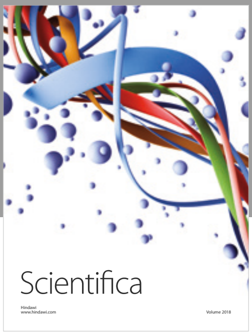
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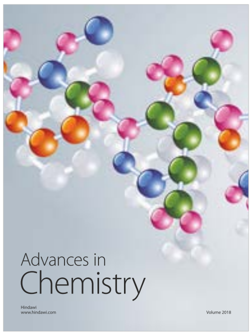
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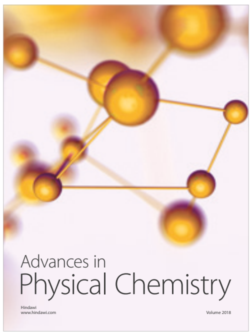
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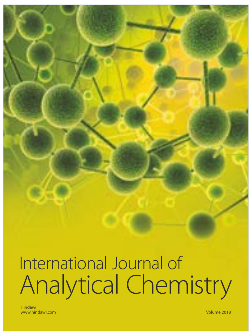
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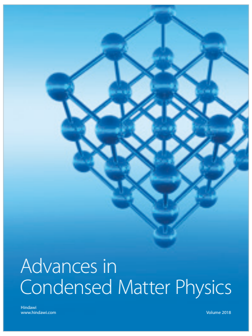
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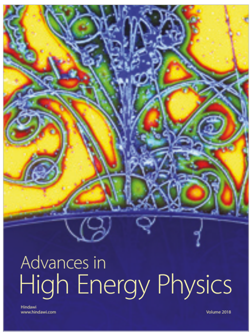
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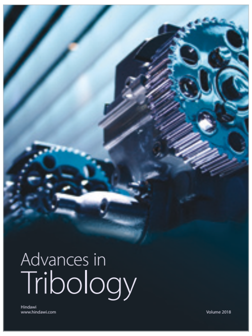
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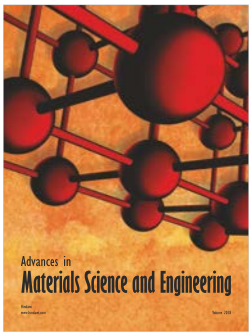
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