

Article

Microstructure and Mechanical Properties of Mg/Al Clad Bars with Ni Interlayer Processed by Compound Castings and Multi-Pass Caliber Rolling

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Abstract: Magnesium/aluminium clad bars were fabricated by compound casting and multi-pass warm caliber rolling. A Ni interlayer prepared using a plasma spraying process was inserted between the parent metals to improve the interfacial characteristics during the casting process, and the effect of caliber rolling on the evolution of the interfacial microstructure and mechanical properties of the Mg/Ni/Al composites was investigated. The results show that the formation of Mg-Al intermetallic phases was impeded effectively by the Ni interlayer and a typical AZ31/Ni/6061 multilayer structure with metallurgical bonding was formed during the compound casting process. In addition, an inhomogeneous strain distribution in the AZ31 and 6061 alloys were characterized during the rolling process. The AZ31 clad layer accommodated a larger proportion of the plastic strain during the initial passes, while the strain in the Mg core layer increased with increasing number of passes. The Ni interlayer fragmented during the rolling process, and transformed into the dispersed particles at the interface. Meanwhile, the fresh AZ31 and 6061 base alloys squeezed out and bonded together under the rolling force, and a well-bonded interface with no visible defects was formed.

Keywords: clad bar; compound casting; caliber rolling; interface; microstructure

1. Introduction

The fast development of modern industries has resulted in the increasing use of lightweight materials in many fields. As important structural materials, magnesium and aluminium alloys are widely employed. The former exhibits low density, high specific strength, and high stiffness, and the latter has good plasticity and excellent corrosion resistance. Clad materials consisting of magnesium and aluminium alloys may be the most effective approach to combine some advantages of the constituent materials that cannot be fully supplied by monolithic materials, and to extend their applications [1–3]. For example, Mg/Al clad materials used in automotive industry can protect from corrosion and reduce the total weight of the automobile, and in turn curb carbon emissions.

To date, various bonding methods have been used to prepare Mg/Al bimetallic composites, including accumulative roll bonding [4], hot co-extruding [5], diffusion bonding [6], friction stir welding [7], explosive cladding [8], high-pressure torsion [9], and compound casting. Compared with other methods, the compound casting process can produce excellent metallurgical bonding between dissimilar alloys at a low cost and a simple production procedure [10,11]. A series of Mg/Al bimetallic composites were prepared using the compound casting process [12–15]. However, the formation of brittle, hard Mg-Al intermetallic compounds in the bonding interface causes an inherent

challenge in bonding the magnesium and aluminium. To improve the interface characteristics of the Mg/Al composites, post-processing with deformation methods appears to be an ideal solution. Paramsothy et al. [16] have reported that no Mg–Al intermetallics were observed in Mg/Al composites fabricated via casting and hot co-extrusion. Wang et al. [17] have used explosive welding and hot rolling to achieve the joining of Al and Mg. Rolling is a widely-used process for fabricating Mg/Al clad sheets, during which the surface oxide layers are cracked and the two fresh base metals are able to contact with each other to form metallurgical bonding under a certain stress. Few reports, however, have been made regarding improving the interface properties of Mg/Al clad bars using the rolling method.

The other well-accepted strategy to improve the bonding strength is to insert a suitable interlayer between the parent metals [18,19]. Ni appears to be a constructive material as the interlayer due to the high melting point and substantial solid solubility in Mg and Al. For example, the wetting and metallurgical bond between the molten magnesium alloy and aluminium substrate were improved effectively by a Ni layer using a “zincate + nickeling” surface treatment [20]. This method, however, is complex and not environmentally friendly. Plasma spraying technology is one of the widely employed surface treatment processes because of low cost, high efficiency, eco-friendly process, and controllable coating on a complex surface. A previous study reveals the Ni interlayer fabricated by plasma spraying can efficiently hinder the formation of Mg–Al intermetallic compounds [21,22]. However, studies of the microstructure evolution of the interlayer during the rolling process are rare, which is important for preparing the clad composites because many as-cast composites need further deformation processing (i.e., clad sheets and rods). In addition, warm caliber rolling is a continuous rolling process used in bar or wire mills for mass production. Up to now, this technique has been mainly used to refine the microstructures of raw materials [23–25], and no study has been reported applying the rolling process to clad materials. The effects that the caliber rolling process has on the interlayer of clad bars have not been clarified to date.

Herein, Mg/Al clad bars were prepared by compound casting with a Ni interlayer, and followed by multi-pass warm caliber rolling. The Ni interlayer was fabricated on the surface of Al substrate by plasma spraying. The interfacial characteristics of the Mg/Al castings with Ni interlayer were studied, and the effects of warm caliber rolling on the microstructural evolution and mechanical properties of the composite castings were also investigated. The findings of this study can play a guiding role in further processes involving clad rods with high efficiency and low cost.

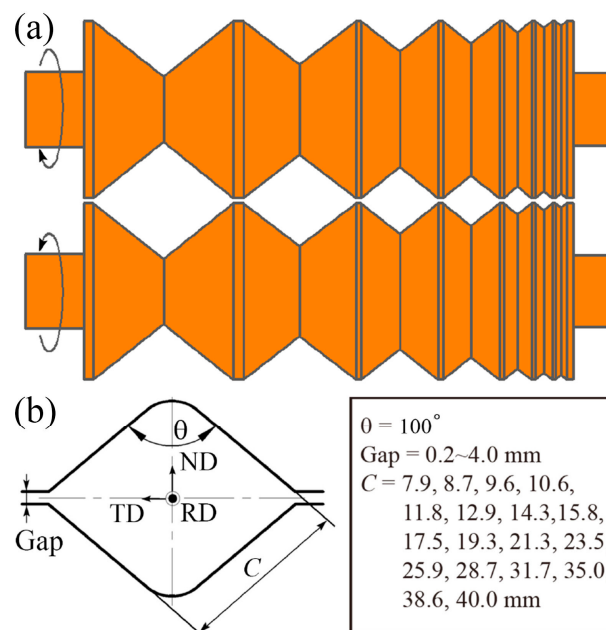
2. Materials and Methods

The matrix materials used in this study were commercial AZ31 magnesium alloy and 6061 aluminium alloy with the chemical compositions given in Table 1. Commercial Ni powders (99.8% in purity) with a mean diameter of 40 μm were used for plasma spraying. The initial AZ31/6061 composites with Ni interlayer were prepared using the compound casting process. First, 6061 specimens with dimensions of 10 mm $\times$ 10 mm $\times$ 100 mm were ground and cleaned with the ethanol solution. The Ni powders were deposited on the surface of 6061 alloy to form a compact layer using plasma spraying technology under the following process conditions: current of 500 A, power of 35 kW, primary gas (Ar) flow rate of 40 L/min, secondly gas (H_2) flow rate of 4 L/min, spraying distance of 100 mm. Then, the 6061 specimen with Ni-coated layer as the insert was mounted inside a stainless steel cylindrical mold with a 36 mm diameter. Finally, the AZ31 alloy was melted and poured into the mold at 690 and 720 $^{\circ}\text{C}$ under vacuum conditions. After solidification, the AZ31/6061 composites with the Ni interlayer were obtained.

Table 1. Chemical compositions of AZ31 and 6061 used in the experiment.

Alloys	Element Content (wt. %)						
	Al	Zn	Mn	Si	Cu	Fe	Mg
AZ31	2.91	0.88	0.42	0.01	0.01	0.02	Bal.
6061	Bal.	0.2	0.13	0.48	0.15	0.58	0.92

The two-high caliber rolling simulator used in the present study and a schematic illustration of caliber rolling is shown in Figure 1. The roll groove comprising a square from 40 to 7.9 mm. The as-cast AZ31/6061 composite was preheated at 360 °C for 1800 s to homogenize the temperature and was then rolled with grooves from 31.7 to 19.3 mm square with no lubrication. Then the bar was cut into two sections. The first was used for testing and the other was rolled to 11.8 mm square under the same initial rolling conditions. The process was repeated to fabricate bars that were passed through 7.9 mm square. Between passes, the sample was preheated at 360 °C for 180 s, and after the final pass the sample was air-cooled to ambient temperature.

**Figure 1.** Schematic illustrations of (a) caliber rolling used for the AZ31/Ni/6061 composite and (b) groove shape.

Metallographic specimens were cut from the AZ31/6061 composites using an electrical discharge machine (MS-430, RONYAN, China), ground with silicon carbide paper, and polished with an alumina polishing solution. Microstructures of the interface of the AZ31/6061 composites were observed using a field emission gun scanning electron microscope (SEM, JSM-7100F, JEOL, Tokyo, Japan). The localized chemical composition at the interface was detected by energy-dispersive X-ray spectroscopy (EDS). To analyse the phase composition at the interface of the as-cast Mg/Al composite, the sample was cut firstly along vertical direction by wire-electrode cutting to obtain a “clad sheet” with dimensions of 10 mm × 5 mm × 60 mm. And then the clad sheet was broken off under a shear force along the bonding interface. The fracture surface was analyzed using X-ray diffraction (XRD, D8 DISCOVER, Bruker AXS, Karlsruhe, Germany). A HMV-2T Vickers hardness tester (SHIMADZU, Kanagawa, Japan) was employed to measure the microhardness of the AZ31/6061 composites with an indentation load of 100 g for 15 s. For each specimen, at least five points were tested to obtain a mean value with a standard deviation error. In addition, the analysis points were spaced so as to eliminate the effect of neighbouring indentations.

3. Results

3.1. Microstructure and Microhardness of the As-Cast Composites

Figure 2a shows SEM image of the interfacial microstructure of the as-cast AZ31/6061 composite with Ni interlayer with a pouring temperature of 690 °C. A uniform Ni interlayer without any obvious reaction layer and defect (i.e., gap) can be observed between the two base alloys. The thickness of the Ni interlayer varied from 7.7 to 32 µm in different locations due to manual operation of the spraying gun, and the average value was 16 µm. In Figure 2b, a higher magnification image of the location highlighted in Figure 2a reveals that a discontinuous intermetallic compound layer with a thickness of about 1.2 µm was formed at the Al/Ni interface. According to the Al-Ni phase diagram (Figure 3) [26] and the EDS result in Table 2, the intermetallic phase was identified as Al₃Ni. By contrast, no visible intermetallic phase was observed along the Mg/Ni interface. The elemental distribution of Al, Mg, and Ni elements across the interface of the as-cast composite are shown in Figure 4. It can be seen that the Mg content as well as Al decreased sharply across the Mg/Ni and Al/Ni interfaces. In the Ni interlayer, however, both Mg and Al elements were not detected, indicating that the interdiffusion between AZ31 and 6061 base alloys was successfully restrained by the Ni interlayer. As a result, the thick and brittle Mg-Al intermetallic phases were absent in bonding interface, which is beneficial to improve the joint performance.

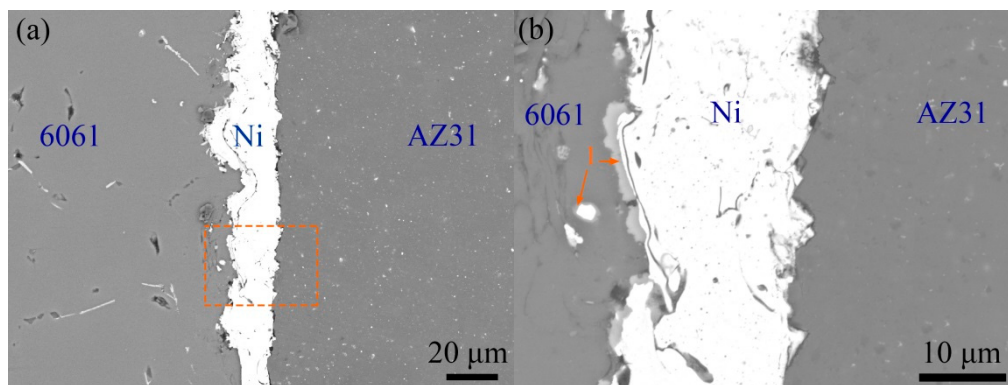


Figure 2. (a) Scanning electron microscope (SEM) image of AZ31/6061 composite castings with Ni interlayer for casting temperature of 690 °C and (b) high-magnification SEM micrograph of highlight region shown in (a).

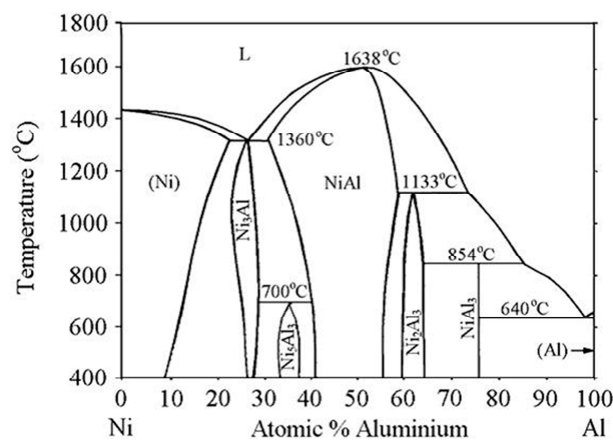


Figure 3. The Ni-Al binary phase diagram [26].

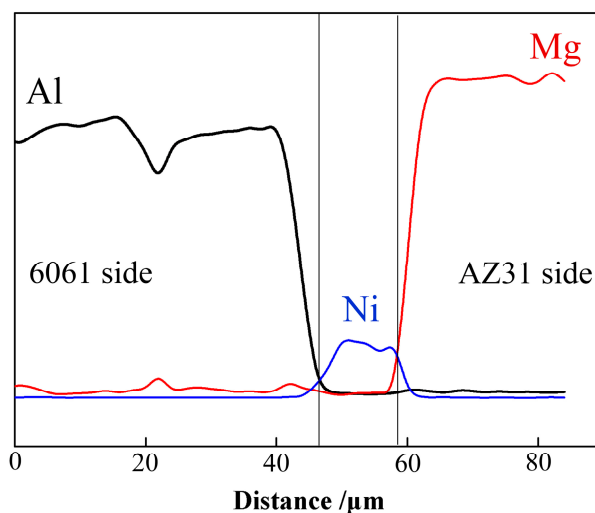


Figure 4. Elemental distribution of Al, Mg, and Ni across the interface of as-cast composite.

X-ray diffraction patterns of the constitutive phases on the fracture surface of the AZ31/6061 composite casting at 690 °C are shown in Figure 5. It is clear that only Ni and Mg were detected on both Mg and Al sides, indicating that the fracture mainly occurred at the AZ31/Ni interface. In addition, although it was detected using SEM and EDS techniques, no obvious pattern of the Al-Ni intermetallic phase was observed. The result also confirms that the Ni inlayer impeded effectively the interdiffusion of Al and Mg atoms across the interface to form intermetallic phases.

Table 2. Energy-dispersive X-ray spectroscopy (EDS) results of the highlighted regions shown in Figures 2 and 6.

Spectrum	Percentage Composition (at. %)		
	Mg	Al	Ni
1	–	75.11	24.89
2	70.95	29.05	–
3	58.46	34.34	7.2
4	2.37	85.48	12.16
5	39.97	60.03	–

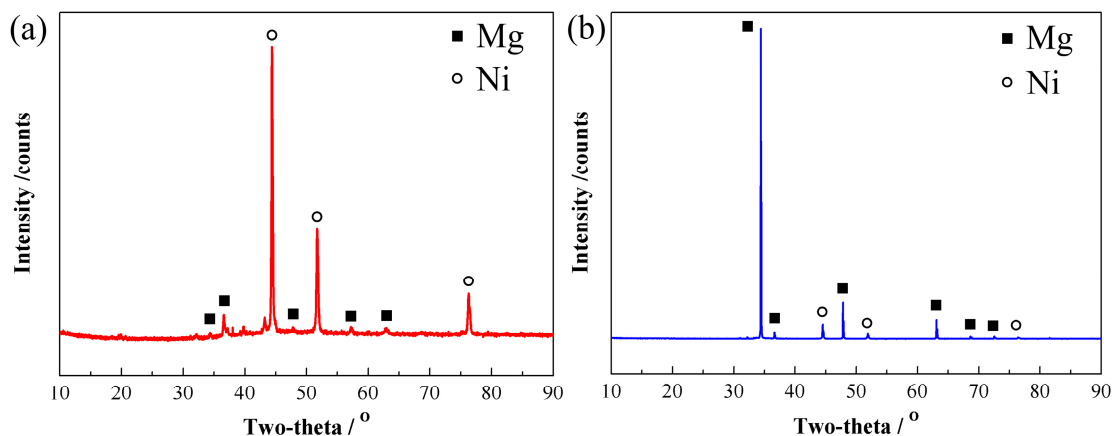


Figure 5. X-ray diffraction (XRD) patterns of fracture surfaces of AZ31/6061 composite castings with Ni interlayer at (a) Al side and (b) Mg side.

During the compound casting process of Mg/Al composites, the pouring temperature is of importance to obtain a good metallurgical bonding between the different alloys. Thus, the effect

of pouring temperature on the microstructure of AZ31/6061 composite with Ni interlayer was investigated. As can be seen from Figure 6a, Ni metal completely reacted with the base alloys to form a reaction layer with a thickness of approximately 500 μm , and the as-sprayed continuous Ni layer was absent with a pouring temperature of 720 $^{\circ}\text{C}$. According to the EDS results (Table 2) and the phase diagrams [26–28], the layers adjacent to the AZ31 and 6061 base alloys were characterized as eutectic structure ($\text{Al}_{12}\text{Mg}_{17} + \text{Mg}$) and intermetallic phase Al_3Mg_2 , respectively, whereas the middle layer was characterized as complex Mg-Al-Ni intermetallic compounds maybe including Al_3Ni , $\text{Mg}_{17}\text{Al}_{12}$, and Mg_2Ni . The elemental distribution in Figure 6b also reveals that there is a clear diffusion zone between AZ31 and 6061 alloys, in which the Mg and Al elements diffuse into each other across the interface, whereas the Ni element diffuses to both base alloys. The Mg-Al-Ni intermetallic compounds were formed with a thickness of approximately 200 μm .

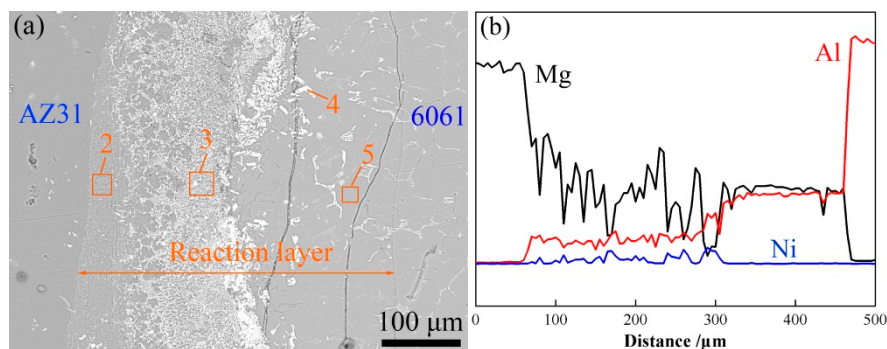


Figure 6. (a) SEM image of AZ31/6061 composite with Ni interlayer for casting temperature of 720 $^{\circ}\text{C}$, and (b) Elemental distribution across the interface. The numbers in (a): spectrum number of regions for EDS analysis.

Figure 7 shows the hardness distribution measured perpendicular to the interface of the as-cast AZ31/6061 composite with Ni interlayer (pouring temperature 690 $^{\circ}\text{C}$). As seen, the microhardness of the Ni interlayer was obviously higher than those of two base alloys. The mean microhardness values of AZ31 and 6061 alloys were approximately 63.8 HV and 73.1 HV, respectively, whereas the mean microhardness value of the Ni interlayer was approximately 155 HV. Nevertheless, the hardness of the Ni interlayer was obviously lower than that of Mg-Al intermetallic phases, which can reach to a value of more than 250 HV [16,22,29].

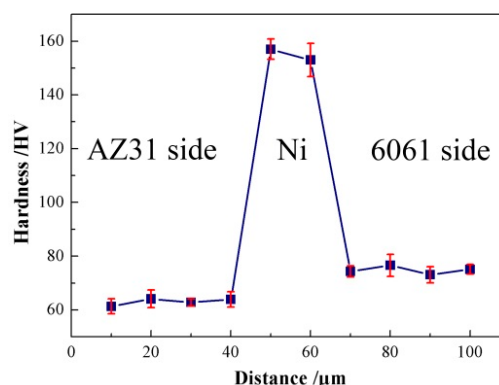


Figure 7. Vickers hardness of AZ31/6061 composite castings with Ni interlayer.

3.2. Characterization after Warm Caliber Rolling

Figure 8 shows a photograph of the AZ31/6061 composite bars after the specimens were passed through square grooves of 31.7 to 19.3 mm (i.e., six passes; “6-pass”), 11.8 mm (i.e., 11 passes; “11-pass”), and 7.9 mm (i.e., 15 passes; “15-pass”). It can be seen that the AZ31 alloy uniformly clad the 6061

alloy core of each sample, and no significant cracking was observed. The casting shows a cylinder shape with the diameter of 35 mm, and the final rolled composite reveals a regular diamond shape on cross-section as expected. It should be noted that the length of the rolled sample could be increased by changing the dimension of the initial compound casting.

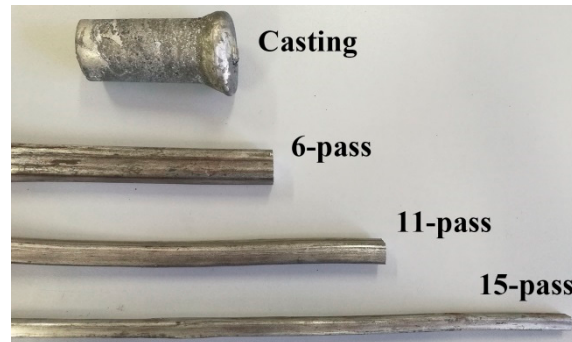


Figure 8. Photograph of the AZ31/6061 composite bars after different numbers of rolling passes.

To investigate the coordination of the deformation that the AZ31/6061 composites undergoes during the caliber rolling process, the relative deformation of the AZ31 and 6061 base alloys was explored. Figure 9 shows the variability of the reduction ratio of the constituent alloys and the clad ratio after different numbers of rolling passes. The reduction ratio is defined as the ratio of the cross-sectional areas of the constituent alloys after and prior to rolling. As seen, the reduction ratio of the AZ31 clad layer increased gradually with the number of passes until ultimately reaching a maximum value of 95.6% after 15 passes. The deformation of the 6061 core layer, however, exhibited a different trend. The reduction ratio of 6061 alloy was much smaller after six passes than that of AZ31, indicating that the AZ31 layer accommodated a larger proportion of the plastic strain. With further increasing number of passes, the reduction ratio of the 6061 core increased quickly to its final value after 15 passes of 95.8%, which was similar to that of AZ31 clad layer. Clad ratio is defined as the cross-sectional area ratio of AZ31 to the total sample. One can see that the clad ratio decreased with the rolling passes up to 11 passes, after which it began to increase. The clad ratio of the final rolled sample was similar to that of the initial as-cast sample.

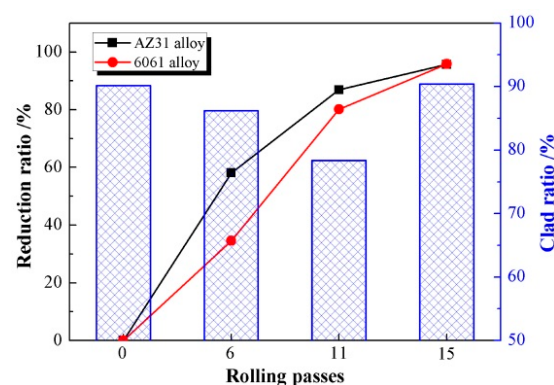


Figure 9. Reduction ratio and clad ratio of AZ31/6061 composite as a function of rolling pass number.

3.3. Microstructural Evolution of Interface

Figure 10a–c shows SEM images of the AZ31/6061 composite bar along the rolling direction after different numbers of rolling passes. As shown in Figure 10a, after six passes the uniform Ni interlayer in the as-cast rod was fragmented and the AZ31 and 6061 base alloys were in contact in some interfacial regions. The fragmented Ni particles seem to be nonuniform. The maximum value of the particle size somewhere in the interface was more than 40 μm , which is larger than the thickness

of as-spray Ni interlayer. One possible reason for this phenomenon is the aggregation of some small fragmented particles under the complex shear stress and compressive stress during the caliber rolling process. When the composite bar was rolled with 11 passes, the fragmented Ni particles became uniform. It should be also noted that 6061 base alloys wrap the Ni particles in some regions and more areas of AZ31 and 6061 bases were bonded together (Figure 10b). With the further increase of the number of rolling passes to 15, the Ni interlayer was nearly eliminated and a well-bonded interface with no obvious intermetallic compounds was formed between the AZ31 and 6061, as shown in Figure 10c. The element distribution across the interface of the final rolled sample is shown in Figure 10d. The Mg content decreased from the AZ31 alloy side to the 6061 alloy side while the concentration distribution of the Al was opposite, indicating interdiffusion of the Al and Mg atoms and a good metallurgical bonding.

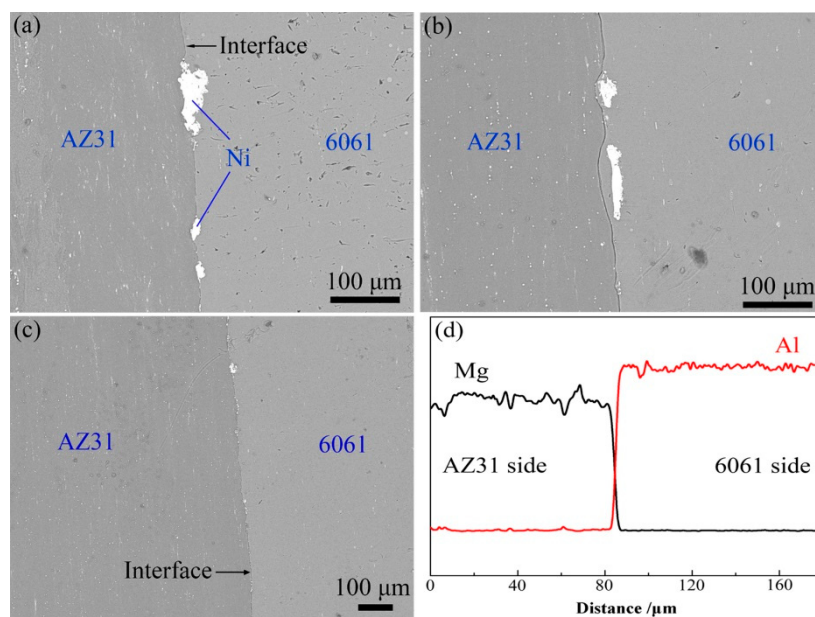


Figure 10. SEM images of the interface of AZ31/6061 composite after (a) 6-pass, (b) 11-pass, and (c) 15-pass rolling. (d) Elemental distribution across the interface.

3.4. Microhardness Evolution of Caliber Rolled Composites

Figure 11 shows the average hardness values of the AZ31 and 6061 layers after different numbers of rolling passes. The microhardness in both layers increased with the increase in strain. After 6-pass rolling, an obvious increase in hardness value was observed in the AZ31 layer while the hardness of the 6061 layer exhibits little change, which may be attributed to the nonuniform deformation mentioned above. The deformation induced a large amount of dislocation in AZ31 layer for work hardening. As the number of passes increases, the hardness value of the AZ31 and 6061 layers increase gradually owing to strain hardening and the grain refinement process. However, the hardness of Mg layers increased slowly with further increased rolling passes. It is supposed that parts of dislocation were recovered in the subsequent preheating process and thus the hardness could not increase rapidly in the further. The final rolled specimen (i.e., 15-pass) exhibits microhardness values of 96.4 and 102.2 HV in the AZ31 and 6061 layers, respectively. It is observed that there is no saturation in the microhardness values of the hybrid composite.

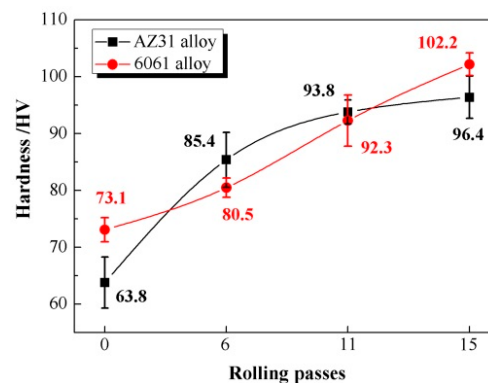


Figure 11. Hardness variation of AZ31 and 6061 layers of Al/Mg composite as a function of rolling pass number.

4. Discussion

It is shown in Figure 2 that the Al_3Ni intermetallic layer with a thickness of about $1.2\ \mu\text{m}$ was observed along the Al-Ni interface. During plasma spraying process, the temperature of the droplet decreased to about $1400\ ^\circ\text{C}$ when it reached the surface of the substrate due to the long spraying distance (100 mm) and small powder diameter (around $50\ \mu\text{m}$). Then the droplet was quenched by the substrate with the temperature of about $150\ ^\circ\text{C}$ and was solidified with the solidification rate of $10^5\sim 10^6\ \text{K/s}$. In addition, the spraying time was less than 10s in this study. Thus, no obvious reaction took place and the good metallurgical bonding was obtained between the Ni droplet and 6061 substrate. During the compound casting process, however, the heat of the AZ31 melt causes the increase of temperature of Ni-coated layer on the 6061 substrate. The atomic diffusion across the interface increased under the condition of high temperature and compositional gradients, resulting in the formation of Al-Ni intermetallics. It should be noted that no visible intermetallic phase was formed at the Mg/Ni interface. One possible reason is that the oxide layer formed at the surface of the Ni layer and AZ31 melt during casting process hindered the interdiffusion of Ni and Mg atoms [30]. Thus, it can be concluded that the Al_3Ni intermetallic layer was generated during casting process rather than plasma spraying process.

As shown in Figure 6, a reaction layer with a thickness of approximately $500\ \mu\text{m}$ was formed in the interface with the pouring temperature of $720\ ^\circ\text{C}$, suggesting that the pouring temperature has a great effect on the interfacial microstructure and diffusion is the dominant mechanism for mass transport [20,31]. With increasing the pouring temperature, interdiffusion of Ni, Mg, and Al were improved. The thinner interlayer was dissolved firstly and formed a diffusion channel, through which Al and Mg atoms diffused to the counterpart easily and formed brittle Mg-Al intermetallic compounds, which will seriously deteriorate the joint performance as reported [19,21,32]. However, It should be noted that the degree of the reaction between the Ni interlayer and the base alloys was also influenced by the thickness of the interlayer. If the layer was thick enough (i.e., about $100\ \mu\text{m}$), it could be difficult to react with base alloys completely even at high temperature [22].

The results of the relative deformation of AZ31 and 6061 base alloys in Figures 8 and 9 show that the strain in AZ31 and 6061 base alloys was inhomogeneous during caliber rolling process. On the one hand, the strain introduced by warm caliber rolling tends to concentrate around the outer part of the sample at the beginning, and was introduced into the center with further increasing the number of passes [33]. On the other hand, differences in mechanical properties of two dissimilar alloys also led to non-homogeneous strain distribution [34].

Furthermore, during the warm caliber rolling process, the mismatch in the plastic deformation between the AZ31 clad layer and the 6061 core layer resulted in a localized shear stress along the interface. Thus, the Ni interlayer, in which some microcracks were formed due to the high-temperature and high-speed conditions during the plasma spraying, cracked and fragmented under this shear

stress. In addition, the compressive stress perpendicular to the interface during the rolling process also increased the fragmentation of the Ni layer. As a result, the hard Ni interlayer was broken and transformed into the dispersed particles, and the fresh AZ31 and 6061 base alloys were squeezed out and bonded together under the rolling force (Figure 10). Zhao et al. [35] have pointed out that the thickness of the reaction layer has a significant effect on the bonding quality of the composite. If the thickness of the Mg-Al intermetallic layers at the interface is greater than 5 μm , the high brittleness and internal stress of the layers would lead to a significant decrease of the ductility and strength of the joints. If the thickness of the layers was less than 5 μm , however, they would have little or no effect on the mechanical properties of the Mg/Al composite. Thus, it can be concluded that the warm caliber rolling process results in the fragmentation of the Ni interlayer and a good metallurgical bonding between AZ31 and 6061, and in turn improves the bonding strength of the AZ31/6061 composite bars. Additional work should be performed to optimize the microstructure of the bonding interface, and further investigate the effects of dispersed Ni particles on the microstructural evolution and mechanical properties of as-rolled composites during post processing.

5. Conclusions

In this work, The AZ31/6061 clad bars were prepared by compound casting process with the Ni interlayer and then were multi-pass warm caliber rolled. The microstructural evolution and mechanical properties of the composites were studied, and the main conclusions are presented in the following:

- (1) The formation of Mg-Al intermetallic phases was impeded effectively by the Ni interlayer, and a well bonded AZ31/Ni/6061 multilayer structure was formed at the interface during the compound casting process. Increasing the pouring temperature resulted in the reaction between Ni and base alloys and the formation of a thick reaction layer composing of complex Mg-Al-Ni intermetallic compounds.
- (2) The strain in the AZ31 and 6061 base alloys was inhomogeneous during the caliber rolling process, mainly being concentrated on and around the clad AZ31 alloy during the initial passes, and subsequently introduced into the center with increasing number of passes. The deformation of the AZ31 and 6061 alloys tended to be equivalent after 15-pass rolling.
- (3) The continuous Ni interlayer fabricated by plasma spraying was fragmented in the subsequent rolling process. With increasing strain, the Ni particle fragments became smaller and uniform. Meanwhile, the fresh AZ31 and 6061 base alloys squeezed out and bonded together under the rolling force. After 15-pass rolling, the hard Ni interlayer transformed into the dispersed particles at the interface and a well-bonded interface with no visible defects was formed.

Results of this study are significant in the preparation of Mg/Al composite bar with high efficiency and low cost. Although some favorable results were achieved, the fragmentation mechanism of the Ni interlayer during rolling process is unclear. Additional work should be performed to control the fragmented process of Ni interlayer and particle distribution during rolling, and to further study the effect of dispersed Ni particles on the properties of Mg/Al clad materials.

Author Contributions: conceptualization, N.L. and C.Y.; methodology, Y.F. and F.Y.; formal analysis, C.L.; investigation, L.C., Y.F., and N.L.; writing—original draft preparation, L.C.; writing—review and editing, N.L.; funding acquisition, N.L.

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Conflicts of Interest: The authors declare no conflict of interest.

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