Microstructure and properties of LZ91 magnesium alloy processed by asynchronous accumulative roll bonding

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Abstract
In order to expand the application prospects of LZ91 magnesium alloy, improve its tensile strength and yield strength. In this paper, the asynchronous rolling and cumulative rolling technology are combined in the large plastic deformation technology, and the LZ91 magnesium alloy after coating the aluminum foil by the asynchronous accumulative roll bonding is performed. The analysis shows that the asynchronous accumulative roll bonding technology accumulates a large amount of deformation energy storage inside the material through large plastic deformation, and refines the grain to the micro-nano level, which increases the strength of the LZ91 magnesium alloy by 86% and the hardness by 63.97%. Improve the internal structure and application performance of the material.

1. Introduction

Li is the lightest metal material on earth, with a density of 0.534 g cm\(^{-3}\), which is about one third of Mg. And the density of Mg is 1.738 g cm\(^{-3}\), which is about two-thirds of aluminum and two-fifths of titanium. It is also an ultra-light metal material [1]. Adding lithium to magnesium alloy will not only reduce the density of magnesium alloy, but also transform magnesium alloy from a hexagonal closed packed (hcp) crystalline structure to a body-centered cubic (bcc) structure [2]. In addition, the Mg-Li series alloy also has advantages of high elastic modulus, high specific strength and specific rigidity, good plasticity and impact toughness, insignificant anisotropy, insensitivity to notches and good damping performance, high penetration resistance to high-energy particles, etc. However, its tensile strength and yield strength are lower than other structural materials, and its corrosion resistance is poor, which severely limits the application of magnesium-lithium alloys [3]. If the severe plastic deformation method can be applied to the preparation of magnesium-lithium alloy sheets, then the aluminum foil is coated during the rolling process, and the comprehensive mechanical properties of the magnesium-lithium alloy are improved by grain refinement.

Accumulative roll bonding technology (ARB) was originally developed by Y Saito [4] et al proposed a process for preparing large-sized ultra-fine grain materials using ordinary rolling mills in 1999. The cumulative stack rolling process breaks through the limitation of traditional rolling reduction [5]. Compared with other Severe Plastic Deformation (SPD) technologies, the advantage of ARB is that the main equipment required for the preparation process is only the rolling mill commonly used in industrial production. It is easy to operate and can continuously prepare thin plates. The shape of ultrafine crystal materials [6], the technology is widely used in aviation, aerospace, automotive important structural fields [7]. A large number of studies on cumulative rolling have recently emerged, mainly in three areas. Using asynchronous accumulative roll bonding technology to prepare multi-layer structure of ultra-fine grain materials, W Jun-li [8] studied the structure evolution and annealing process of ultrafine-grained copper; C Kwan [9] and others studied the cumulative lamination of aluminum alloys and found that the oxides at the welding interface can prevent the grain boundaries from welding. Migration at the interface; G Krallics et al [10] cumulatively rolled ultra-low carbon steel at 500 °C, and

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2. Experiment

the strength and plasticity of the ultra-low carbon steel increased. 2 Using asynchronous accumulative roll bonding technology to prepare multi-metal layered composite materials. A Mozaffari [11] cumulatively rolled aluminum and nickel and found that the cumulative strain of the aluminum layer was more; Eizadjo, cumulatively rolled with M copper and aluminum found that copper would eventually break up and be dispersed into the green collective. 3 Use asynchronous accumulative roll bonding technology to prepare layered structure particle reinforced metal matrix composites. Mohammad [12] to study the effect of accumulative roll bonding on the combination of reinforced TiO2 and the matrix. The presence of TiO2 nano-particles (even at very low content) increased the peeling force of aluminum strips significantly.

Asynchronous rolling technology (AR) is one of the large plastic deformation technologies. Originally, in the early 1940s, when Germany was studying single-roller-driven lamination sheet and the Soviet Union was studying a three-roller Lauter mill [13], the asymmetry of the circumferential speed of the two rolls produced a unique shear of the rolled material in the deformation zone thin plate strip processing technology evolved from deformation conditions. Asynchronous rolling technology can reduce rolling pressure and improve sheet processing efficiency [14]. Lv Aiqiang et al [15] found that the asynchronous rolling can effectively improve the structure orientation of the aluminum foil surface layer through the study on the transformation of the aluminum foil texture by asynchronous rolling. S J Liang et al [16]. After asynchronous rolling of Mg–Al–Zn alloy, micron-level fine-grained alloy was obtained.

Severe plastic asynchronous accumulative roll bonding technology (AARB) is a new technology method that combines accumulative roll bonding technology and asynchronous rolling technology [17]. The Severe plastic asynchronous accumulative roll bonding technology not only increases the equivalent strain of the material during the rolling process, but also increases the shear stress asynchronously on the basis of accumulative roll bonding, to break up the refined grains and improve the comprehensive mechanical properties of the material [18]. AARB combines the advantages of asynchronous rolling and accumulative roll bonding technology. The metal in the deformed area of the asynchronous rolling can be subjected to additional shear stress, and the rolling pressure can be reduced when the reduction is equal. The larger equivalent strain during conventional rolling and grain refinement can control the basal texture orientation in the alloy to a certain extent [19]. AARB can make two metal sheets of equal size superimposed together and make them automatically welded, and then repeat the same process to repeat the stacking and welding, resulting in a layered structure, so that the material gets a large cumulative true strain, therefore, the structure is refined and the mechanical properties of the material are greatly improved. While increasing the layer structure of the material, the grains are refined, and the internal structure of the material is improved. This paper combines asynchronous rolling and accumulative roll bonding technology in large plastic deformation technology, and performs asynchronous accumulative roll bonding on aluminum foil coated with LZ91 magnesium alloy to prepare high-performance LZ91 magnesium alloy strip.

| Element | Li | Zn | Ni | Fe | Cu | K | Na | Mg |
|---------|----|----|----|----|----|---|----|----|
| Content | 9.05 | 1.05 | 0.002 | 0.002 | 0.0005 | 0.004 | 0.004 | Bal. |

Table 1. Chemical composition of LZ91 magnesium alloy for experiment (wt%).

In this experiment, the magnesium–lithium alloy strip (LA91) provided by Chinalco Zhengzhou Light Metal Research Institute was used with a thickness of 0.5 mm. And its chemical composition is shown in table 1.

Cut the hot-extruded LA91 magnesium alloy sheet into a strip specimen of 60 mm * 35 mm * 0.5 mm by wire cutting, and then heat the specimen in a vacuum tube furnace at 200 °C for 5 h annealing to obtain the original structure. Polish the magnesium board with a wire brush until it is bright to remove the oxide layer on the surface of the magnesium board, and then clean the surface with acetone to remove the surface oil. The two pieces of surface–treated magnesium alloy sheet are aligned up and down, with a layer of aluminum foil in between, and a layer of aluminum foil is wrapped after one end is riveted. Asynchronous accumulative roll bonding is carried out on the two-high asynchronous rolling mill LG-300, the diameter of the upper roller is 124 mm, the diameter of the lower roller is 134 mm, and the asynchronous ratio is 1:1.08. During rolling, the roll shaft is not heated and not lubricated. Before each pass, the rolled sample is heated in a box-type resistance furnace at 250 °C for 5 min, and then the sample is subjected to a single pass asynchronous accumulative roll bonding with a reduction of 50% Cumulative rolling is carried out 4 passes respectively. The samples with different rolling passes were named AARB0, AARB1, AARB2, AARB3, AARB4. Grind the sample on the metallographic sandpaper, and then polish and corrode, use Olympus BX51M metallographic microscope to
observe the microstructure and take pictures. Corrosion agent metallographic corrosion agent: oxalic acid 2 g + nitric acid 1 ml + absolute ethanol 97 ml. Cut the thin sample block with wire cutting, mechanically grind the sample to thin it, and then punch it into a disc with a diameter of 3 mm. Use an ion thinner to thin and perforate. After making the TEM sample, it is placed on the GTF30S-Twin transmission electron microscope. Observe. At room temperature tensile test was carried out on the samples of asynchronous accumulative roll bonding 4 passes, and the tensile strength and elongation of the tensile samples at room temperature were measured. VEGA 3 SBH scanning electron microscope was used to observe the microstructure and fracture morphology of the material, and the observation surface was the rolling surface. The equipment selected for XRD analysis is D/max-Rc X-ray diffractometer. X-ray diffractometer working parameters: Cu target, tube voltage 40 kV, tube current 40 mA, scanning step length 0.01 0, scanning rate 2 deg.min-1. A sample is taken from the cross section of the laminated composite board, and the hardness of the sample is measured by the HSX-1000A microhardness tester. The load used is 0.1 N, and the interface is kept for 10 s for multiple tests. Take 5 reliable values and calculate average value.

3 Results and discussion

3. Experimental results and analysis

Figure 1 shows the metallography of different passes, from left to right are four passes, three passes, two passes, one pass and the original state. It can be seen from figure 1 that the original structure after annealing is thick. According to the binary phase diagram and the metallographic diagram of the magnesium-lithium alloy, it can be determined that it is mainly a two-phase alloy composed of the magnesium phase (α phase) and the lithium phase (β phase), and it is mainly composed of the white base magnesium alloy phase and the gray second-phase lithium phase [20]. The β phase has more slip systems, and the plastic deformation ability is relatively good. The coarse α phase is evenly distributed in the magnesium-lithium alloy, the grains are about 100 to 200 microns, the phase interface is clear, and the two phases are distinct. It can be seen from the metallographic pictures of samples that are asynchronous accumulative roll bonding in one pass, after one pass, α phase begins to be flattened and widened, and the area increases, and α phase and β phase are elongated along the rolling direction during the rolling process. In the metallographic picture of the sample after two asynchronous accumulative roll bonding, the α phase accumulates stress with the increase of the amount of deformation, and it is further broken and refined during the rolling process, the grain is refined and further uniformly dispersed to β phase. The number of large-sized grains in the metallographic diagram of the samples after three times of AARB is reduced,
and the grain size of the magnesium-lithium alloy is further refined to separate the $\beta$ phase more uniformly. It can be seen from the metallographic diagrams of the samples after four-pass AARB that after four-pass AARB, a large amount of strain is accumulated, making the grain fully refined. The $\alpha$ phase is also fully flattened along the rolling direction, and the fracture is broken and evenly dispersed into the magnesium-lithium alloy matrix, so that no obvious $\alpha$ phase can be found in the metallographic picture.

Figure 2 shows the microstructures diagram of the rolling surface of the fourth pass asynchronous accumulative roll bonding. It can be seen from the figure that the thickness of aluminum layer decreases to less than $10 \mu m$ when the rolling passes are increased to four passes. The increase in the amount of deformation makes the grains of the aluminum layer elongated, broken, and refined, and the thickness of the aluminum layer is reduced or even broken. With the fracture of the aluminum layer, different layers of magnesium alloys continue to appear at the bonding interface. The grains of the magnesium layer and the aluminum layer, the

Figure 3. Distribution of elements on RD-ND surface of the fourth-pass asynchronous accumulative roll bonding sample.

Figure 4. TEM microstructure of asynchronous accumulative roll bonding LZ91 sheets in different passes: (a) AARB1; (b) AARB2; (c) AARB3; (d)–(f) AARB4.
magnesium layer and the magnesium layer are continuously squeezed, rubbed, and bonded at the interface to make the interface welded. The aluminum layer was severely thinned after four passes of asynchronous cumulative rolling, and even fractured and dispersed into the magnesium matrix. Because aluminum has 12 slip systems, and there is no reinforcing phase in the aluminum layer, the aluminum layer deforms greatly during rolling. At the bonding interface, due to the crushing of the aluminum hard block, the magnesium-lithium alloy layers are in contact with each other and welded together.

Figure 3 shows the scanning chart of the RD-ND surface energy spectrum of the asynchronous accumulative roll bonding of the fourth pass. It can be seen that the interface of magnesium and aluminum after four passes of rolling is clear, the interface is tightly combined, and the interface between magnesium and aluminum is clean without other impurities, except for oxygen elements. The apparent oxygen content at the interface is due to the oxidation of the interface in contact with air during the rolling process. A large part of the oxygen content of the energy spectrum scan is caused by the oxidation of the sample surface during the preparation of the scan sample. It can be seen from the Fig that the magnesium-lithium compound is slightly oxidized, and oxygen only gathers near the aluminum layer, and no oxygen penetrates into the magnesium matrix. It shows that the oxidation of magnesium-lithium alloy is well suppressed during the processing, and the reliability of asynchronous cumulative rolling is increased. On the other hand, the aluminum foil coating not only protects the magnesium lithium alloy and improves the corrosion resistance, but also has excellent electrical conductivity. The addition of aluminum layer can improve the conductivity of the matrix, which can broaden the application field of magnesium lithium alloy.

Figures 4(a)–(e) shows the transmission diagram of the AARB samples of different passes, and (f) is a dislocation diagram. It can be seen from figure 4 that the sample after large deformation and AARB contains a large amount of dislocation accumulated in the stress contrast and distributed around the \( \alpha \) phase. Figure 4(a) clearly shows that the grains are flattened and elongated along the rolling direction, the grain size is above 500 nm, and there are a lot of dislocations inside the \( \beta \) phase, but not inside the \( \alpha \) phase. It can be seen from figure 4(b) that the density of dislocations in \( \beta \) phase further increases, and the accumulation of a large number of dislocations increases the stress contrast in the transmission diagram. It can be clearly seen from Fig that the crystal grains are obviously refined at about 400 nm. In figure 4(c), the grain refinement is obviously around 200 nm, and the \( \alpha \) phase and \( \beta \) phase are evenly dispersed, and the dislocations are evenly distributed with the \( \beta \) phase. The grain refinement in figure 4(d), (e) is obviously below about 100 nm, which is the dynamic recrystallization of nano-scale fine crystals. In figure 4(e), the area with small dislocation stacking density is selected, and the diffraction spot taken by the grain is selected, and it is calibrated as \( \alpha \) phase grain.

Finally, in figure 4(f), it can be seen that the internal dislocation density of the sample increases after asynchronous accumulative roll bonding. Due to the small cubic structure of the \( \alpha \) phase slip system, it is relatively difficult to deform, and the sever plastic asynchronous accumulation during buckling deformation, when the dislocation slip deformation encounters a large \( \alpha \) phase, the slip stops. Dislocation accumulation in the \( \beta \) phase before the \( \alpha \) phase increases the dislocation density and increases the internal energy. During the rolling process, dynamic recrystallization is prone to occur to reduce the internal energy of the matrix, so that the grains will be fine during the rolling process. However, there are some magnesium-lithium alloy phase slip systems far
away from the second phase, which are relatively easy to start slip, and the dislocation density is relatively small, which makes the grain change little. Therefore, the small grains that are broken and dynamically recrystallized during the rolling process are about a dozen micron, while the coarse grains without refinement are still about 100 microns, resulting in grain structures of different sizes. Fine grains and a large number of dislocation tangles are the two reasons for the improvement of the mechanical properties of the material.

Figure 5 shows the XRD spectrum of magnesium-lithium alloy by asynchronous cumulative rolling bonding. It can be seen in the Fig that the alloy is mainly composed of two phases of Li_{0.92}Mg_{4.08} and Li_{3}Mg_{7}. During the rolling process, aluminum foil is coated, so that the Al phase appears in the XRD spectrum of one, two, three or four passes. By comparing the PDF cards, we know that Li_{0.92}Mg_{4.08} is a close-packed hexagonal structure, and Li_{3}Mg_{7} is a bcc structure. According to the binary phase diagram, LZ91 magnesium alloy is mainly composed of $\alpha$ phase and $\beta$ phase, namely Li_{0.92}Mg_{4.08} is $\alpha$ phase and Li_{3}Mg_{7} is $\beta$ phase.

Figure 6(a) shows the tensile test results of the large plastic asynchronous accumulative roll bonding specimen. It can be clearly seen that after rolling, the plasticity of the specimen is significantly reduced, the tensile strength is significantly improved, and with the increase of the number of rolling passes, the plasticity gradually decreases in turn, and the tensile strength gradually increases. The work hardening of the sample due to large deformation and asynchronous accumulative roll bonding reduces the plasticity of the sample and increases the tensile strength. In the course of the rolling process of the sample, the hard $\alpha$ phase is first flattened and elongated in the rolling direction, and while being continuously broken, it is evenly dispersed into the matrix. Due to the pinning effect of the hard phase ($\alpha$ phase), the $\beta$ phase slip is not easy to proceed, forming a dislocation cluster. It can also be seen from figure 4 that the refined grains of $\beta$ phase recrystallization are always around the $\alpha$ phase, and recrystallization always nucleates at the defect or interface, thus inferring the hard $\alpha$
phase during the asynchronous cumulative lamination process. A large number of dislocations are formed around it for the dynamic recrystallization of $\beta$ phase, and the refinement of $\beta$ phase will also increase the strength of the sample. On the other hand, when the sample is subjected to asynchronous cumulative lamination below the recrystallization temperature, a large amount of plastic deformation, slipping of the crystal grains, dislocation entanglement occurs, the grains are elongated, broken and fibrillated, and a large amount of accumulated inside the material Residual cold work hardening phenomenon. Furthermore, the tensile strength of the sample increases and the plasticity decreases. And with the increase of the asynchronous cumulative lamination pass, the harder the $\alpha$ phase is refined, the more uniform the dispersion, the better the cold work hardening and fine grain strengthening effect.

Figure 6(b) shows the relationship between asynchronous accumulative roll bonding pass and microhardness. From the microhardness graph, it can be seen that the hardness of the sample increases with the increase of asynchronous cumulative lamination passes. From the original annealed state of 49.05HV, it gradually increased to 80.43HV of the asynchronous cumulative rolling four passes, and the microhardness increased by 63.97%. During the asynchronous cumulative rolling, the internal structure of the magnesium-lithium alloy strip has been effectively refined. Under the action of the rolling force, the grains are gradually broken, the distortion energy continues to increase, and the internal dislocations in the alloy continue to increase. The increase further hinders the plastic deformation of the alloy, so that the deformation resistance inside the alloy gradually increases and the hardness increases. From table 2, it can be seen more intuitively that the tensile strength and microhardness values of the LZ91 magnesium alloy both increase in sequence with the increase of cumulative asynchronous lamination passes. After four passes, the tensile strength increased by 86% and the microhardness increased by 63.97%.

Figure 7 shows the tensile fracture morphology of different asynchronous accumulative roll bonding passes. It can be seen from the Fig that the fracture is flat without large fluctuations, indicating that the $\alpha$ phase is evenly dispersed in the matrix after asynchronous cumulative rolling, even after annealing, there is no phenomenon of $\alpha$ phase aggregation. According to the tensile test results, it can be seen that the strain after the asynchronous cumulative rolling is significantly reduced, and there is no obvious necking near the fracture. It is inferred that the samples after asynchronous cumulative rolling are mainly brittle fracture. It can be seen from the fracture morphology that, except for figure 7(a), the other fractures are a large number of tear ridges and smooth quasi-cleavage fracture zones, and only a small number of small dimples are dispersed in the fracture along the rolling direction. And with the increase of asynchronous accumulative roll bonding passes, the number of dimples decreases and becomes smaller, and the fracture morphology is dominated by quasi-cleavage fractures. Mainly due to the extension of the hard $\alpha$ phase along the rolling direction during the rolling process, a large amount of stress is accumulated around the hard $\alpha$ phase, so that the hardness of the matrix increases and the plasticity decreases, cleavage fracture occurs, and it is extremely easy to stretch. A cleavage fracture source is generated at the $\alpha$ phase hard spots and intracrystalline dislocation defects. Due to the continuous dynamic recrystallization of the magnesium matrix during the rolling process, a fine-grained magnesium matrix is distributed around the hard $\alpha$ phase, and a small number of fine dimples will be generated during the stretching process.

### 4. Conclusion

1. Sever plastic asynchronous accumulative roll bonding can effectively refine the grains. The LZ91 magnesium alloy after four-pass rolling can obtain micron to nanometer grains. The refinement of the grains effectively improves the mechanical properties of the LZ91 magnesium alloy.

2. After sever plastic asynchronous accumulative roll bonding, the LZ91 magnesium alloy contains a lot of dislocation tangles, which improves the hardness and strength of the LZ91 magnesium alloy.

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**Table 2. Mechanical properties of samples with different rolling passes.**

| Samples | Tensile strength (MPa) | Elongation (%) | Microhardness (HV) |
|---------|------------------------|----------------|-------------------|
| AARB0   | 117                    | 26.43          | 49.1              |
| AARB1   | 161                    | 14.06          | 55.4              |
| AARB2   | 173                    | 9.67           | 61.9              |
| AARB3   | 192                    | 6.27           | 72.2              |
| AARB4   | 219                    | 4.29           | 81.5              |

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