Microstructures and electromagnetic interference shielding effectiveness of ME21/Mg laminated materials by accumulative roll bonding

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Abstract

Laminated composite with multi-layer interfaces has better electromagnetic interference shielding performance, which has attracted great attention. In this work, magnesium matrix laminated structure materials were prepared through Accumulative Roll Bonding (ARB). Microstructure, electrical conductivity and electromagnetic interference (EMI) shielding effectiveness (SE) of ME21/Mg laminated materials were investigated to understand the effect of layered structure and the change of microstructure on the electromagnetic shielding property. The results showed: the precipitated secondary phase and introduced interfaces could provide multiple reflections, attenuate the electromagnetic waves and improve the SE value. The electrical conductivity of 2-cycle increased to $21.04 \times 10^6$ S m$^{-1}$, which was 17.74% higher than that of ME21 alloy, the intensity of texture of ME21 layer increased with the rolling passes, which contributed to the improvement of the electrical conductivity as well as the attenuation of reflection. The layered composite exhibited better shielding effectiveness compared with the ME21, in the 8.2–12.4 GHz test frequency, the SE was 98–107 dB. The shielding mechanism of layered materials was explained, which provided guiding for the efficient shielding of electromagnetic waves.

1. Introduction

Electronic noise as well as electromagnetic pollution has been verified being harmful to not only human health, but also surrounding equipment [1, 2]. As the increase of electronic devices and the rapid development of communication technology, mobile terminals and base stations have a lot of incremental demand for electromagnetic shielding products [3]. Millimeter wave propagation in the fifth-generation (5G) applications has a very high demand for electromagnetic interference (EMI) shielding. In addition, large amount of heat will be generated due to the high integration and propagation speed of 5G [4, 5]. Electromagnetic shielding and heat conduction products have also ushered in a huge growth space. Thus, it is significant to develop light weight and integration of structure and functional materials to alleviate EMI pollution.

Copper, silver and nickel are traditional metal shielding materials because of their good conductivity [6, 7]. However, high density, high cost and difficult processing hinder its application in the industrial field. Polymer matrix composites have light density and good corrosion resistance, they have weak electromagnetic shielding performance. The coatings on polymer have good conductivity, which can effectively attenuate the shield electromagnetic waves. But the coating is easy to be scratched and then lose its electromagnetic shielding property, meanwhile the poor heat conductivity of the polymer materials restricts the development in the field of civil [8, 9].

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Magnesium and its alloy have the advantages of light weight, high specific strength, excellent damping, as well as good heat conduction properties and electrical conductivity [10], which are regarded as the potential electromagnetic shielding materials. Song et al obtained different intensity of basal textures of AZ31 by varying the strain, which amplified the impedance mismatch between the external free space and sheets, the SE value was improved by the increase of reflection attenuation [11]. Some researchers reported that the electromagnetic shielding performance of magnesium is general in the low frequency band [12, 13]. The magnesium alloy with secondary phases will promote the multi-reflection and then attenuate the electromagnetic wave. Gao et al studied the different rolling and heat treatment on the Mg-xSn alloy electromagnetic interference shielding performance, and found that the precipitation of secondary-phase on the basal plane improved the shielding effectiveness (SE), by the way of multiple reflection losses [14]. Chen et al explored the as-cast Mg-Y-Zr alloy with different Nd contents, the EMI shielding capacity was enhanced with the amount of Nd via the precipitation of the secondary phases [15].

ME21 alloy is a new type of magnesium alloy which is usually used in automobiles and communications fields. It has good mechanical properties and corrosion resistance [16]. The rare earth phase in the ME21 alloy will increase the electromagnetic shielding performance through multi-reflection. However, the SE of multi-reflection loss is relatively low, so we need to improve the SE of multiple reflection by building a layered structure through the accumulative roll bonding process [17].

Accumulative roll bonding (ARB) is a severe plastic deformation process, which was proposed by Saito et al [18, 19]. In the rolling process, the process not only imposes high plastic strain on the material, but also promotes diffusion bonding of the adjacent interfaces, which is suitable for the preparation of laminated sheet composite materials. Wang et al explored the ARB process on the electromagnetic interference shielding performance of the Mg-9Li-3Al-1Zn, and found the shielding effectiveness increased gradually with the ARB passes, which was attributed the alternative arrangement of α-Mg and β-Li phase and introduced layers [20]. Hou et al studied the electromagnetic shielding property of duplex magnesium alloy, after rolling for 4 passes, the materials exhibited the best shielding performance of 73–84 dB, in the test frequency of 30–1500 MHz [13]. However, up to now, there are few reports on the preparation of layered composites by processing magnesium alloys with different components by ARB process, and fewer references reported that the laminated composites was applied to the field of electromagnetic shielding.

In this work, the layered composites of pure magnesium and ME21 magnesium alloy were prepared by ARB process, and the rolling pass on the microstructures, electrical conductivity and electromagnetic shielding property was investigated, the study will expand the application of layered materials in electromagnetic shielding industry.

### 2. Materials and process

The fabrication process of the Mg/ME21 layered composite material was shown in figure 1. The four main procedures were included. (1) The surfaces of the sheets were treated by sandpaper, until the surface was smooth; (2) the plates were stacked in the order of ME21, Mg and ME21; (3) the accumulated sheets were rolling by the six-roller mill; and (4) the rolled plates were cut into equal parts and then rolled again. ME21 magnesium alloy and pure Mg were processed with the dimensions of 150 × 100 × 1 mm, the chemical composition of ME21 alloy is listed in table 1. Before rolling, the rollers were preheated to the 270 °C by oil. At first, the cumulative plates were rolled by the 50% reduction without lubricant, and the thickness of the sheets reached to 1.5 mm, and then continued to roll until the thickness of the composite sheets reached to 1 mm, such plates were named ‘sandwich-pass’. The sheets were then rolled in two passes, named ‘1-cycle’ and ‘2-cycle’ respectively. Before each ARB cycle, the surfaces were cleaned with the acetone, and then removed the oxide layer and other impurities with the sandpaper to ensure surface roughness and sufficient bonding strength. The plates were annealed at 300 °C for 1 h in the furnace before the next pass of rolling. Finally, laminated composites with 12 layers were obtained.

| Table 1. Chemical composition (wt%) of ME21 alloy. |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Mg | Mn | Ce | Fe | Ca | Si | Al | Zn | Ni |
| 97.30 | 1.83 | 0.76 | 0.045 | 0.003 | 0.006 | 0.02 | 0.018 | 0.002 |

Microstructure observations were examined using an optical microscopy (OM) and a scanning electron microscope (SEM) equipped with EDS. The composites were examined by electron backscattering diffraction (EBSD), the samples for EBSD orientation mapping were prepared by mechanical grinding and electrochemical...
polishing by electrolyte, the EBSD dates were analyzed by the Channel 5 software. The phase determination was carried out with x-ray diffractometer (XRD). The electrical conductivity of the sheets was measured with Sigma 2008B conductivity meter, and each conductivity value was the average of five tests. The standard coaxial cable method in accordance with ASTM D4935–2010 was used to determine the EMI SE in the X-band (8.2–12.4 GHz), the samples with the dimensions of 41.4 × 41.4 × 1.0 mm.

3. Results and discussion

3.1. Microstructure of laminated composite materials
Figure 2 displayed the macro-graph of the interfaces in the sandwich pass. The surface of pure magnesium had many waves and large deformation, which indicated that the deformation of pure magnesium was large.

The three-dimensional optical micro-graphs of the structure of sandwich-pass and 2-cycle were shown in figure 3. In the sandwich-pass, the thickness of each layer was basically the same, the compactness of the interface bonding was not enough. With the increase of ARB passes, the ME21 alloy and pure Mg formed the alternating layered structure, the number of interfaces increased, the warm rolling process promoted the metallurgical diffusion bonding of the interfaces, the interfaces were more closely combined simultaneously. Due to the different plastic deformation capacity, in order to keep the uniform deformation of the plate, the
interfaces presented the wave structure, each layers had different deformation, and neck shrinkage, fracture and other phenomena.

Figure 4 presented the SEM images of the composite materials with different rolling passes, and found that in the sandwich-pass, the interfaces were relatively flat, some parts were closely bonding, and some parts had poor bonding phenomenon, such as holes and cracks. As shown in the figure 4(b), in the 1 cycle rolling process, due to the welding of similar interfaces, the interfaces could be well combined under the process condition of 50% reduction, and the bonding effect was very excellent. In addition, the composite interfaces of ME21 and Mg were improved, and the combination of ME21 and Mg was closer, the holes almost disappeared and the interfaces presented a continuous compounding state. However, it could also be seen that because the plastic deformation capacity of these two materials was very different, the phenomenon of necking and uneven deformation occurred in the process of deformation. With the increase of ARB passes, in order to coordinate the deformation of the sheets in the overall deformation process, the uneven deformation of the inner layers became more serious, and some parts were fractured, the interface bonding of ME21 and pure Mg were more tightly.

The SEM images of the alloy and composites were illustrated in figure 5. There were many particles with different sizes and different shapes scattering in the ME21 alloy layers inhomogeneously. The amount of
secondary phase increased with the increase of rolling passes, and the precipitates were much finer. Owing to the ARB rolling adopting the warm rolling process, in addition, the material generated heat during large deformation, after two passes of rolling, the more secondary phases of Mg12Ce and Mn precipitated from the matrix, and the secondary phases of Mg12Ce grew up during deformation and distributed in the matrix, which was consistent with the XRD patterns (figure 10). Figure 6 displayed the interface bonding of 2-cycle, and the interfaces were very well integrated. In the figures 6(b) and (c), the results of EDS showed that the secondary phases of Mg12Ce and Mn precipitated at the interfaces, which enhanced the strength of the interfaces, which was similar to the mechanical welding process.

Figure 7 illustrated the grain orientation of ME21/Mg with different passes. In the figure 7(a), the layer interfaces were clear, the left of the layer interface was ME21 layer, the right was Mg layer. Compared with the sandwich-pass, dynamic recrystallization occurred in the composites after ARB for 2-cycle, the coarse grains decreased, the fine equiaxed grains increased, and the microstructure became uniform. Texture evolution of pure Mg layer of ME21/Mg composite materials under ARB process was shown in figure 8, which was typical rolling texture, the max texture intensity of the structure of sandwich-pass was 28.55, the intensity of orientation changed to 16.41 after ARB for 2-cycle, the texture intensity decreased slightly, in figure 4(c), after ARB for 2-cycle, the 'wave' structure was formed in the magnesium layers, which leaded to the weaken of the component.
deformation texture, and a large number of fine recrystallized grains led to the decrease of texture [17]. Figure 9 illustrated the texture evolution of ME21 layer of ME21/Mg composite materials under ARB process. The texture was typical rolling base texture (0001), after the initial rolling, the intensity of orientation was 19.88, and the orientation intensity increased to 48.22 with the ARB for 2-cycle, which indicated that the c-axis of most
Figure 9. Texture evolution of pure ME21 layer of ME21/Mg composite materials under ARB process.

Figure 10. XRD patterns of different alloys.
grains was perpendicular to the rolling surface [11]. In addition, the texture deflected to the rolling direction, because the shear force caused the texture of sheets to deflect to the rolling direction in the process of ARB process [17].

XRD patterns of different alloy was shown in figure 10. With the increase of ARB passes, the diffraction peak intensity of Mg12Ce and Mn phase enhanced, which showed that ARB process was helpful to the precipitation of the secondary phases in the preparation of composite materials. X-ray diffraction showed the phases in the composites, the results indicated that no evidence of new intermediate compound formation was found.

3.2. Electrical conductivity

Figure 11 showed the electrical conductivity of the different materials. The electrical conductivity of ME21 and the sandwich–pass was $17.87 \times 10^6$ $\text{S m}^{-1}$, $20.54 \times 10^6$ $\text{S m}^{-1}$, respectively. The electrical conductivity was greater than that of sandwich–pass after ARB 1-cycle. The reason was that large reduction deformation reduced interface holes, in addition, the secondary phase precipitation was induced at the interfaces, which promoted the interface bonding, the good interface combination enhanced the electrical conductivity. With the increase of ARB passes, the change of electrical conductivity tended to increase. The electrical conductivity of 2-cycle increased to $21.04 \times 10^6$ $\text{S m}^{-1}$, which was 17.74% higher than that of ME21 alloy. In the figure 4(c), after 2-cycle, the ME21 was more tightly bonded to the Mg, which constructed into a layered structure. The electrical conductivity of Mg was 23.5% higher than that of ME21, the layered structure was analogous to a material that electrical conductivity well filling a material that electrical conductivity poorly. The resistivity increased with the amount of alloying elements in solid solution [21]. Conductivity depended on exonuclear electrons, the solute atoms dissolving in the matrix would cause serious lattice distortion and become the center, which scattering electrons and phonon, as a straightforward understanding, changing the direction of motion of electrons and phonon [22]. Under the synergistic action of warm rolling process and large deformation, the secondary phase precipitated dynamically and grew up. With the increase of rolling passes, the number of secondary phases also increased, meanwhile, the solute atoms dissolving in the matrix decreased, which reduced the obstruction of the free electrons and phonons, and profited to the improvement of electrical conductivity. In the figure 9, the texture of ME21 layers became stronger with the increase of ARB passes, magnesium metal had a dense hexagonal structure, and the resistivity along c-axis was smaller than that along a-axis, resulting the increase of the conductivity [14, 23].

3.3. Electromagnetic shielding properties

Figure 12 illustrated the EMI SE of the different materials. In the test frequency of X-band, the EMI SE curve depicted the wavy. The SE values of sandwich–pass, 1-cycle, 2-cycle were 91–103.5 dB, 94–104 dB, 98–107 dB, respectively, as shown in figure 12(a), which exhibited good shielding capacity. Usually, the SE of civil industry requirement is 30–60 dB and the SE of military requirement is 60–120 dB. Correspondingly, the SE value of the pure Mg and ME21 were 78–99 dB, 97–103 dB. In the figure 12(b), the average SE value of composites increased from 97 dB to 101 dB. With the increase of ARB passes, the SE of the composites increased, after 2-cycle, the SE was best, which the change trend was consistent with the electric conductivity.

The electromagnetic shielding property after ARB for 2-cycle was greater than that of 1-cycle, because the number of interfaces increased inside the material after compounding. The shear deformation also occurred on
the internal layer due to the action of shear stress, so that the interfaces formed the wave structure, which helped to improve the multi reflection ability and formed more alternating layered structures inside the material.

Shielding is to ‘cut off’ the transmission path from electromagnetic interference source to electronic equipment, to reduce the impact of interference source on equipment [24]. The EMI shielding mechanism diagram was shown in figure 13, which was better to understand the EMI shielding intuitively. When electromagnetic waves incident into the materials, according to the equation (1), the electromagnetic waves will be influenced by four different shielding mechanisms, including the reflection (R), absorption (A), multiple reflection (M), and circular reflection (C). The transmission line analogy was adopted to explain electromagnetic shielding of plane waves by Schelkunoff [25, 26]. According to Schelkunoff theory, reflection (SE_R), absorption (SE_A) and multiple reflections (SE_M) constitute to the EMI SET, which is the total shielding effectiveness.

\[
SE_T = SE_R + SE_A + SE_M + SE_C
\]

\[
SE_R = 168.2 + 10 \log \left( \frac{\sigma_c}{f \mu_c} \right)
\]

\[
SE_A = 131.43 \sqrt{f \mu_c \sigma_c}
\]

\[
SE_M = 20 \log \left( 1 - e^{-2 \delta_c} \right) = 20 \log \left( 1 - 10^{\frac{SE_A}{10}} \right)
\]

\[
SE_C \propto N
\]

\[
\delta_c = \sqrt{\frac{1}{\pi \mu_c \sigma_c f}}
\]
where, $\mu_r$ is the relative magnetic permeability, $\sigma_f$ is the relative electrical conductivity, $f$ is the frequency of electromagnetic waves (Hz), $t$ is the thickness of the shielding materials (m), $\delta_f$ is the skin depth (m), $N$ is the the number of the interfaces for circular reflection.

The electromagnetic shielding mechanisms were depicted in figure 13. The incident electromagnetic waves are reflected due to the impedance mismatch between the shielding materials and the external environment, with the increase of conductivity, $SE_R$ is enhanced. Due to the relative dielectric constant of the shielding material, when the electromagnetic wave hits the shielding material, the charge will be induced internally, resulting in an electric field to offset the electric field in the incident electromagnetic wave, resulting in the attenuation of the electromagnetic wave. With the increase of the frequency of the incident wave, the absorption ($SE_A$) loss increases. When the residual electromagnetic waves penetrate into the layered composite material, at the overall level of the material, the shielding efficiency of absorption is mainly composed of dielectric loss and magnetic loss of alternately distributed magnesium layers and ME21 alloy layers. At low frequency, the electromagnetic wave energy is weak, and most of the incident electromagnetic wave will be reflected by the surface of the alloy. However, at high frequency, the electromagnetic wave energy often penetrates the metal [27]. The secondary phase and multi-layer structure inside the alloy play an important role in the attenuation of electromagnetic wave [21], moreover, the structure of the laminated sheets is special geometric structure, with the increase of ARB passes, more interfaces will be formed inside the sheet, the lamellar distribution in the plate is not uniform, therefore, the $SE_M$ exerts on the attenuation of electromagnetic waves. Since the electrical conductivity of ME21 and magnesium is different, the impedance of adjacent layers is inconsistent, in the beginning, electromagnetic waves pass from the outside into the outermost ME21 layer, and then pass from the ME21 layer into the magnesium layer, because of impedance mismatch, will attenuate the intensity of the electromagnetic wave by the way of reflection at the interfaces, similarly, when electromagnetic waves travel from the magnesium layer to the ME21 layer, reflection loss occurs again due to inconsistent impedance, following this pattern, the electromagnetic wave in the shielding materials will occur many times such circular reflection attenuation, according to the equation (5), the attenuation is proportional to the number of layers with inconsistent impedance. During the warm rolling process, a large amount of secondary phases precipitate in the ME21 sheets, owing to the impedance mismatch between the secondary phases and the surrounding matrix [28], electromagnetic waves will be reflected and attenuated at the interface between the matrix and the secondary phase, the secondary phase can scatter electromagnetic waves inside the plate. As a result, the electromagnetic waves can be reflected multiply between the interfaces of ME21/Mg laminated sheets and the secondary phases.

4. Conclusions

The ME21/Mg composites were prepared successfully by ARB process, it has efficient shielding of electromagnetic waves. Following conclusions could be drawn:

1. In the ARB process, due to the different plastic deformation ability, the alternating layered structure had different deformation, and the ARB process promoted the bonding of the interfaces. There were the precipitations of the secondary phase at the interface, which promoted the bonding strength of the interfaces.

2. After ARB 2-cycle, in the test frequency range of 8.2–12.4 GHz, the EMI SE of the layered composite materials reached to 98–107 dB, the electrical conductivity was $21.04 \times 10^6 \text{ S m}^{-1}$, compared with the ME21 alloy. In addition, the density of the layered composite material decreased. The solid solution elements in the ME21 matrix formed the secondary phase precipitations, reducing the scattering of electrons and increasing the electrical conductivity, which increasing the reflection loss. The introduced interfaces and the secondary phases increased the attenuation of multi-layer reflection.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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