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Microstructure, mechanical and corrosion properties of magnesium alloy bone plate treated by high-energy shot peening

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Abstract: To enhance the mechanical properties and corrosion resistance of magnesium alloys, high-energy shot peening (HESP) was used. According to the results, the in-situ surface nanocrystallization (ISNC) microstructure was fabricated on the magnesium alloy surface, and its formation mechanism was the coordination among twins, dislocations, subgrain boundary formation and dynamic recrystallization. Under the released surface stress of sample, the residual compressive stress and microhardness rose, thus enhancing compactness of the surface passivation film Mg(OH)₂. Besides, the corrosion rate dropped by 29.2% in maximum. In the polarization curve, the maximum positive shift of the corrosion potential of sample was 203 mV, and the corrosion current density decreased by 31.25% in maximum. Moreover, the compression resistance and bending resistance of the bone plate were enhanced, and the maximum improvement rates were 18.2% and 23.1%, respectively. Accordingly, HESP significantly enhanced mechanical properties and corrosion resistance of magnesium alloys.

Key words: magnesium alloys; high energy shot peening; in-situ surface nanocrystallization; mechanical properties; corrosion properties

1 Introduction

Magnesium and magnesium alloys have aroused rising attention in the medical fields of cardiovascular scaffolds, bone plates and orthopedic implants for their similar density and elastic modulus for natural bone, biodegradability, excellent mechanical properties, biocompatibility as well as tissues adsorption [1-3]. However, compared with the conventional inert metal biomaterials (e.g. stainless steel and titanium alloys), magnesium and magnesium alloys are still flawed. The reason for this is that they are susceptible to corrosion and rapid degradation in the human body, they can generate a large amount of hydrogen gas, and their mechanical properties are low [4,5].

To enhance the mechanical properties and corrosion resistance of magnesium alloys, many measures have been taken (e.g. alloying, microstructure modification and surface modification) [6–8]. Among them, the alloying method can to some extent achieve the required

corrosion resistance and mechanical properties, whereas the biocompatibility and corrosion behaviors of magnesium alloys remain unsatisfactory due to the impurities effect [9]. Surface modification cannot only reduce the rate of degradation, but also enhance biocompatibility of magnesium alloys. However, the weakness of interface adhesions results in poor mechanical properties of magnesium alloys, thereby significantly limiting their potential applications in biomedical field [10]. Besides, microstructure modification is now the commonest method, including the use of powder metallurgy method, severe plastic deformation (SPD) method and rapid solidification method [11-13]. To be specific, the SPD is a candidate method used to achieve ultra-fine grain structure and enhance mechanical properties of magnesium alloys [14]. However, the improvement of corrosion resistance of magnesium alloys by the SPD method has been rarely reported.

Recently, during the process of surface modification, a protective nanopassivation film adhered to the matrix

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tightly has been proven an effective method to enhance corrosion resistance, which is named ex-situ surface nanocrystallization (ESNC), whereas it cannot enhance mechanical performance of magnesium alloys [15,16]. Moreover, since using the SPD method can form an ultrafine grained structure on the surface of the material, the method is also termed as in-situ surface nanocrystallization (ISNC) [17]. Thus, it is preliminarily considered that the ISNC process can enhance both mechanical properties and enhance corrosion resistance. Among them, high-energy shot peening (HESP) is cheaper, more convenient, and more effective to ultimately achieve ISNC [18]. Though HESP has been proven as an effective and economical way to produce ISNC, and its effects on mechanical properties of magnesium alloys have been systematically studied, the mechanism of ISNC formation by HESP remains unclear [19]. Besides, the corrosion resistance and mechanical properties of magnesium alloys after HESP have been rarely discussed.

Accordingly, in this work, the ISNC microstructure on the surface of WE43 magnesium alloy bone plate was formed through HESP, its potential in improving mechanical properties and corrosion resistance was assessed, and the formation mechanism of ISNC was analyzed.

2 Experimental

2.1 High-energy peening test

The material used in this work was Mg–4%Y– 3.3%RE (Nd,Gd)–0.5%Zr rare earth magnesium alloy sheet (20 mm \times 20 mm \times 10 mm). The peripheral side surface of the sample should be polished orderly by sandpaper down to 2000 grit until the surface became smoother and flatter. The high-energy penning equipment used in this investigation was designed and manufactured by Kaitai Shot Blasting Machinery Co., Ltd., Shandong, China. The schematic diagram of the device and the bone plate shot peening process are shown in Fig. 1. The principle of shot peening was as follows: under the high-pressure gas, the projectile had a certain initial velocity, and a high-speed jet beam was sprayed onto the surface of the workpiece through the airbrush. Large plastic deformation would occur on the surface of the workpiece to cause grain breakage and lattice torsion, thereby resulting in grain refinement and the enhancement of material properties [20]. The almen intensity and surface coverage in the shot peening process were set to 0.4 N and 100%, respectively. The entire shot peening process was split into two steps to strengthen kinetic energy of the shot peening process, in an attempt to induce grain refinement on the top surface layer of the sample. First, the stainless steel ball with a diameter of 0.20 mm was taken as the projectile, and the gas pressure was 0.70 MPa. Besides, the impact distance was 300 mm, the shot angle was about 90°, the processing time was 2 min and 4 min, respectively, and then the samples were named according to the processing time. Step 1 aims to induce severe plastic deformation. On that basis, in step 2, the glass bead with a diameter of 0.15 mm was taken as the projectile, and its hardness was HV 500. The pressure of gas was 0.4 MPa, the impact distance was 100 mm, the shot angle was about 90° and the processing time was 12 min and 15 min, respectively. Step 2 aims to induce the formation of ISNC.

2.2 Mechanical properties test

Microhardness measurements were performed using a MXT70 digital microhardness tester with a diamond Vickers indenter under a load of 100 g for 20 s on three paths starting from the treated surface towards the core material. Each depth region was tested three times to achieve the average value, and the step size was nearly 0.1 mm.

The surface residual stresses of magnesium alloys were measured using a $\sin 2\theta$ method with the X-ray stress analyzer (iXRD, Proto). Moreover, the



Fig. 1 Schematic diagram of high-energy shot peening: (a) Device diagram; (b) Shot peening principle

standardized parameters used for the residual stress measurements are listed in Table 1.

Three-point bending tests and compressive tests were performed at ambient temperature using a Ziess electronic universal testing machine. Besides, the 8-hole straight shaped magnesium alloy bone plates of 140 mm \times 14 mm \times 3.8 mm (Weigao Group Medical Polymer Co., Ltd., Shandong, China) with and without undergoing the shot peening were taken as test samples, as shown in Fig. 2. Besides, to achieve more accurate test results, the loading speeds of 1, 2 and 5 mm/min were adopted in the indenter, and the tests were stopped until the magnesium alloy bone plate was significantly deformed. Moreover, to ensure the repeatability of the bending and compression test curves, all samples were tested three times at different indenter speeds. The specimens of each bone plate could be used only once.

Table 1 Parameters used for residual stress measurements

Parameter	Standardized value
Collimator type	1 mm diameter
X-ray target material	Cu K _a
Peak 20/(°)	156
Exposure time/s	10
β angle/(°)	-45 to 45
X-ray tube voltage/kV	20



Fig. 2 Mechanical properties tests: (a) Three-point bending tests; (b) Compressive tests

2.3 Microstructure characterization

To remove surface contaminants, the shot peened samples were ultrasonically cleaned in ethanol for 10 min. The 3D topography of the surface of the sample was observed under a VH-Z500R 3D ultra-deep digital microscope.

Before the microstructure observation, the corresponding surface polishing should be performed. The samples were ground by 400–1500# sandpapers and subsequently polished by electrolytic polishing

instrument at -25 °C. Finally, the polished samples were corroded by picric acid for 3–5 s. The microstructures of samples before and after being treated were investigated under optical microscopy (OM, Leica DM2000), transmission electron microscopy (TEM, JEOL JEM–2100F) and electron back scattered diffraction (EBSD, HKL Nordlys).

2.4 Corrosion properties test

First, the WE43 magnesium alloy bone plate after shot peening was cut into a standard sample of 10 mm \times 10 mm. Subsequently, the non-working surface of the samples was fully sealed with Kraft silicone rubber. Next, before the corrosion resistance test was started, the samples were fully washed with distilled water and ultrasonically processed in acetone ethanol for 10 min.

The salt spray corrosion test was an accelerated experimental method for the evaluation of the corrosion resistance of metallic materials [21]. The neutral salt spray test was performed using YWX/Q-150 salt spray corrosion test chamber at ambient temperature according to "GB/T10125-1997, artificial atmospheric corrosion test". The corrosion period was taken as 24 h. After 4, 8, 12, 16 and 20 h, respectively, the samples were taken out and weighed after the corrosion products were removed according to the physical method specified by ISO8407 [22]. It is noteworthy that the un-shot peening samples were used as the reference in this work.

The electrochemical corrosion test was performed using Shanghai Chenhua CHI604E electrochemical workstation. The classical three-electrode system was employed in the experiment. The working electrode was the tested samples (the working area was 10 mm \times 10 mm), through complete immersion, the auxiliary electrode was a platinum sheet, and the reference was an Ag-AgCl (in saturated KCl) reference electrode. The test was performed in 0.9 wt.% NaCl solution to simulate the chloride concentration in the human body environment [23]. The potential scanning range was -1.8 V to -1.0 V, and then the Tafel curve scanning rate was subsequently set to 1.0 mV/s after the open-circuit potential (OCP) was steady enough (about 30 min). To achieve one polarization curve with good repeatability, the measurements of all specimens were performed at a constant ambient temperature of 37 °C at least three times.

The corrosion rate was calculated using the mass loss method. After the corrosion test, the surface corrosion morphology of the sample was observed under the scanning electron microscopy (SEM, Philips XL30 FEG), and the product adhering to the surface was characterized using X-ray diffraction (XRD, Bruker D8 Advance). The SEM device was operated with the backscattered-electron detector, and the accelerating voltage and the working distance were 3 kV and 5 mm, respectively. The XRD device was operated under a Cu K_a (λ =0.1541 nm) radiation, the 2 θ range of 10°-80° with a step size of 0.02°, a measurement time per step of 0.2 s, and a Ni filter. To ensure the repeatability of the XRD pattern, all sample tests were performed three times.

3 Results and discussion

3.1 Mechanical properties

3.1.1 Microhardness and residual stress

The microhardness-depth curves of the samples treated with different shot peening time are shown in Fig. 3(a). In this figure, the depth represents the distance from the treated surface to interior of the sample. The microhardness after HESP treatment was significantly enhanced, compared with that of the untreated samples, and the increasing rates were 87.8% (4 min), 105.2% (12 min) and 110.4% (15 min), respectively. Moreover, with the increase in the depth, the microhardness of the samples after HESP treatment gradually decreased. In brief, the surface microhardness of WE43 magnesium



Fig. 3 Mechanical properties of samples treated for different shot peening time: (a) Microhardness and surface depth; (b) Residual stress and surface depth

alloy was significantly improved by HESP and increased as the shot peening time was prolonged.

The residual stresses induced by HESP with different depths are shown in Fig. 3(b). In this figure, the positive and negative values represent tensile stress and compressive stress, respectively. The results reveal that when the depth was less than 0.1 mm, the absolute value of residual compressive stress decreased gradually as the shot peening time was prolonged. Once the depth was greater than 0.2 mm, the absolute value of residual compressive stress would gradually increase. This was because shot peening can lead to stress release on the surface of the sample, resulting in an increase residual compressive stress. In other words, with the increase in depth, the absolute value of residual compressive stress first increases to the maximum, then gradually decreases, and finally converts to the tensile stress inside the matrix. 3.1.2 Bending force and compression force

The curves of the compression and three-point bending tests of different shot peened bone plates with different displacement rates of the indenter are plotted in Fig. 4. The results reveal that different compression load curves (Figs. 4(a), (c), (e) and (g)) and bending load curves (Figs. 4(b), (d), (f) and (h)) have similar variation rules, i.e., as the displacement continued, the compression force first increased and then decreased, while the bending force increased all the time. Besides, as the displacement rate increased, the bending stress increased gradually, while the compressive force decreased gradually. For the compression test, the bone plate exhibited an elastic deformation phase before the yield load point, while after the yield load point, it exhibited a bending deformation phase, and then the plate failed and was deformed permanently. For the bending test, also in the stage prior to the yield load point, the bone plate was elastically deformed; as long as the applied bending load was less than the yield load (P), the deformation of the bone plate would disappear when the bending force was removed. Thus, the yield load (P) acts as a vital indicator to evaluate the strength of the bone plate. The compressive and bending yield loads of the bone plates treated at different shot peening times at three loading speeds of indenter are listed in Tables 2 and 3, respectively. The average compressive yield loads of bone plates were 4163.7, 4299.8, 4577.1 and 4922.1 N, increased by 3.3% (4 min), 9.9% (12 min) and 18.2% (15 min), respectively. Besides, the average bending yield loads of bone plates were 673.3, 717.5, 766.9 and 828.4 N, increased by 6.5% (4 min), 13.9% (12 min) and 23.1% (15 min), respectively. Moreover, both in bending and compression teats, the yield point appeared within the displacement range of 1-2 mm. Thus, the bending resistance and compression resistance of the bone plate after shot peening were enhanced.



Fig. 4 Curves of compression and three-point bending tests of different shot peened bone plates under different displacement rates of indenter: (a, b) 0 min; (c, d) 4 min in step 1; (e, f) 12 min in step 2; (g, h) 15 min in step 2 (P_1 , P_2 and P_3 are the yield loads of the bone plate at the indenter displacement rates of 1, 2, 5 mm/min, respectively)

1 0	1			
Speed of indenter/	Yield load/N			
$(\text{mm} \cdot \text{min}^{-1})$	0 min	4 min	12 min	15 min
1	4530.2	4682.5	4927.2	5240.8
2	4250.5	4377.3	4638.5	5022.4
5	3710.4	3839.6	4165.4	4503.6
Average	4163.7	4299.8	4577.1	4922.1

 Table 2 Yield load of bone plates treated for different shot

 peening time under compression tests

 Table 3 Yield load of bone plates treated for different shot

 peening time under three-point bending tests

Speed of indenter/	Yield load/N			
$(\text{mm} \cdot \text{min}^{-1})$	0 min	4 min	12 min	15 min
1	640.2	687.3	732.6	793.5
2	670.6	713.5	764.8	823.9
5	710.5	751.7	803.4	867.7
Average	673.3	717.5	766.9	828.4

3.2 Microstructure characterization after high-energy peening treatment

3.2.1 Surface characteristics

The 3D morphologies of surfaces treated by shot peening at different steps and different time are shown in Fig. 5. It is noteworthy that the surface profile of the sample started to be uneven when the shot peening time was 2 min (Fig. 5(a)). Moreover, many craters were found on the treated surface. As the shot peening time was prolonged, the number of craters increased, so the surface roughness was enhanced gradually as well. The distance between the peaks and valleys of the surface profile is represented by DPV (marked as red circle). After 2 min of shot peening, the sample had the DPV value of 7.6 μ m (Fig. 5(a)), after 4 min, it was 33.71 μ m (Fig. 5(b)), after 12 min, it was 27.40 μ m (Fig. 5(c)), and after 15 min, it was 24.49 μ m (Fig. 5(d)). This suggests that the DPV increased first and then decreased as the shot peening time was prolonged. The reason for this was that the shot peening coverage of the sample surface gradually increased as the shot peening time was prolonged.

3.2.2 Quasi surface nanocrystallization formation mechanism

Figure 6 shows OM microstructures of the cross section of samples shot peened for 2, 4, 12 and 15 min, respectively. At 2 min (Fig. 6(a)), parts of grains started to form dynamic recrystallization and turn to fine equiaxed grains (marked by red circles). However, some large grains (marked by rectangle boxes) were remained the essential aspects, so the specimen exhibited a nonuniform microstructure. After 4 min of HESP treatment, ultra-fine grains appeared in the plastic deformation layer, some of which were nanocrystalline (grains size <100 nm). This plastic deformation layer is defined as a quasi-surface nanocrystallization (quasi-SNC) layer [24]. The thickness of quasi-SNC layer was about 80 µm, as shown in Fig. 6(b). Over time, the thicknesses of quasi-SNC layers of the samples shot peened were 130 and 110 µm for 12 and 15 min, respectively. The reason for this was that as the shot peening time was prolonged, more surface material was



Fig. 5 3D morphologies of specimen surfaces treated by shot peening: (a) 2 min in step 1; (b) 4 min in step 1; (c)12 min in step 2; (d) 15 min in step 2



Fig. 6 OM images of cross section of samples treated for different shot peening time: (a) 2 min in step 1; (b) 4 min in step 1; (c) 12 min in step 2; (d) 15 min in step 2

removed, resulting in a gradual decrease in the thickness of the quasi-SNC layer, and meantime, the surface average grain size also decreased. Furthermore, some mechanical twins caused by severe plastic deformation were observed on the treated surface. These plastic deformations were caused by many high-speed particles impacting on the material surface in a short period, and partly caused grain refinement. When the shot peening time increased to 12 min, more twins appeared. In the meantime, several low-angle grain boundaries ($2^{\circ}-15^{\circ}$, green lines) were observed, as shown in the insert of Fig. 6(c). When the shot peening time increased to 15 min, more dynamic recrystallized grains (DRXs) were formed, and more pronounced grain refinement was observed, as shown in Fig. 6(d).

Figure 7 displays the EBSD orientation maps of WE43 magnesium alloys in different HESP steps. The deformation mechanism and microstructure evolution after HESP treatment were further revealed by EBSD [25]. The similar color of the grains represents the smaller misorientation angle. Different colors represent different orientations of the grains. Figure 7 suggests that EBSD verifies the correctness of the above OM analysis. In other words, suitable processing parameters of HESP induce formation of nanograins on the surface of the sample. The average grain size of untreated samples was 900 nm, that of step 1 was 80 nm, and that of step 2 was 32 nm, as shown in Fig. 7(d). Figure 8 shows the TEM

microstructures of samples surface after shot peening for 4, 12 and 15 min, respectively. Dislocation tangle and subgrain boundaries phenomena could be observed in the samples shot peened for 4 min (Fig. 8(a)) and 12 min (Fig. 8(b)), respectively. As the shot peening time was prolonged, the deformation of the surface of magnesium increased continuously, alloy and considerable sub-structures (e.g. dislocation tangle and subgrain boundaries) replaced the original twin or lath-like structure. Thus, the grains were gradually refined. Besides, when the actual deformation of the crystal structure exceeded the critical deformation amount, the DRX was induced to form nanocrystalline grains, as shown in Fig. 8(c) [26]. Thus, the above conclusions were further verified.

The schematic diagram of the formation mechanism of the surface nanocrystallization due to HESP is shown in Fig. 9. The blue regions, the dash lines and the fine green grains represent the twins, subgrain boundaries and dynamic recrystallized grains, respectively. First, under the impact of HESP, large plastic deformation would occur on the surface of the samples, leading to occurrence of mechanical twinning. As the twins increased, more grain boundaries hindered dislocation movement, resulting in dislocation entanglement and pile-up. Accordingly, a quantity of subgrain boundaries were formed. When the distortion energy increased as the shot peening time was prolonged, the DRX was



Fig. 7 EBSD orientation maps of WE43 magnesium alloys: (a) Under ingot state; (b) After shot peening in step 1; (c) After shot peening in step 2; (d) Average grain size



Fig. 8 TEM images of samples treated for different shot peening time: (a) 4 min in step 1; (b) 12 min in step 2; (c) 15 min in step 2

induced to form refined grains. Thus, the grain refinement mechanism of the HESP WE43 magnesium alloy was achieved through the coordination of twinning, dislocation rearrangement, subgrain boundaries forming and dynamic recrystallization.

3.3 Corrosion properties

3.3.1 Corrosion morphology and corrosion rate after salt spray corrosion test

The surface corrosion morphologies of untreated and treated HESP samples after salt spray corrosion test are shown in Fig. 10. Due to the rapid corrosion under simulated salt spray conditions, the untreated samples showed large cracks and corrosion pits (Fig. 10 (a)). Besides, after shot peening for a period, only some etch pits were formed on the surface of the samples (Figs. 10(b) and (c)), while after sufficient time treatment, the surface of the samples was uniform and substantially free of etch pits (Fig. 10(e)). The reason for this was that as the shot peening time was prolonged, the pits were rapidly filled by corrosion products (Fig. 10(d)), which might cause the corrosion to slow down, thereby improving the corrosion resistance.

The corrosion degree of untreated and treated HESP



Fig. 9 Schematic diagram of surface nanocrystallization mechanism by high-energy shot peening



Fig. 10 SEM images of sample surface after salt spray corrosion: (a) Untreated; (b) 2 min in step 1; (c) 4 min in step 1: (d) 12 min in step 2; (e) 15 min in step 2

samples are expressed by the corrosion rate. The average corrosion rate of the samples after corrosion was evaluated using the mass loss index [27]. The calculation formula is expressed as

$$v = \frac{m_0 - m_1}{St} \tag{1}$$

where v denotes the corrosion rate, m_0 is the mass of the samples before corrosion test, m_1 is the mass of the samples after removal of corrosion products, S is the area of corroded samples, and t is the corrosion time of samples.

The corrosion rate changes in the salt spray corrosion test of untreated and treated samples are shown in Fig. 11. The results suggest that the corrosion rate of untreated and treated samples decreased rapidly during the time interval of 8 h (marked as region I), while a slow downward trend was observed between 16 h (marked as region II) and 24 h (marked as region III). Accordingly, as the corrosion time was prolonged, the corrosion rate curve eventually was leveled off. Moreover, during the whole corrosion, the corrosion rate of treated sample was lower than that of untreated samples except the region III. In region III, the corrosion rate decreased as the shot peening time was prolonged. In other words, the corrosion rate was $0.017 \text{ mg/(cm}^2 \cdot \text{h})$ after being treated for 15 min while $0.024 \text{ mg/(cm^2 \cdot h)}$ for untreated sample, and the corrosion rate decreased by 29.2%. The reason for this might be that the surface microstructures of the samples were different. Shot peening led to nanocrystallization by severe plastic deformation on the surface layer, and the original coarse grains caused DRX to form fine grains, which led to the increased of grain boundaries. First, the corrosion rate rose as the grain boundaries increased. For instance, two grain boundaries (marked a and b as shown in Fig. 11) could lead to significant variations in corrosion rates. It is suggested that Type a grain boundaries were ingot state while Type b grain boundaries were the boundaries of fine grains after shot peening. Compared with Type a, Type b had more grain boundaries, exhibiting a higher activity and more energy of metal atoms [28]. Thus, Type b grain boundaries tended to suffer more significant corrosion first. However, once the dense films are formed, passivation and protective effects will be dominant, and corrosion rate significantly will decline. Accordingly, HESP treatment leads to the improvement of corrosion resistance of samples.



Fig. 11 Corrosion rate change of samples untreated and treated for different HESP time

Furthermore, magnesium alloy is a type of natural passivation metal, whereas it is very easy to pit, resulting in the passive film being destroyed in a non-oxidizing media encountered Cl⁻ [29]. When WE43 magnesium alloy is in NaCl salt spray media, alloy surface will be oxidized by H⁺, the ion balance on magnesium alloy surface will be destroyed, and the pitting nucleus will occur at the weak structure of grain (e.g. grain boundaries) because of high concentration of Cl⁻ attracted and enriched on the metal surface with rise in the concentration of H⁺. This is because Cl⁻ shows preferential adsorption than oxygen on some active sites, accelerating the corrosion of magnesium alloys. Moreover, the nonuniform microstructure becomes the

nucleus preferentially formed pitting place [30]. Thus, magnesium alloys without shot peening treatment are more susceptible to pitting corrosion, resulting in the increase of corrosion rate. The XRD pattern of corrosion products after shot peening is shown in Fig. 12. By the X-ray diffraction analysis, except that NaCl was not thoroughly cleaned, the main component of product attached onto the surface of material after corrosion was Mg(OH)₂, and meantime, Mg(OH)₂ was combined with Cl⁻ and H₂O molecules to form hydrated Mg₂Cl(OH)₃·4H₂O. Furthermore, the slightly soluble Mg₂Cl(OH)₃·4H₂O reduced the area where surface ions could move freely and hinder the entry of oxygen and other media, thereby protecting the passivation film and slowing the corrosion rate in the later stage [31]. Accordingly, the main corrosion progress of WE43 magnesium alloy in 3.5% NaCl salt spray is expressed in Eqs. (2)–(5).

$$Mg \rightarrow Mg^{2+}+2e$$
 (2)

 $2H_2O+2e \longrightarrow H_2+2OH^-$ (3)

$$Mg^{2+}+2OH^{-} \longrightarrow Mg(OH)_2$$
 (4)

$$2Mg(OH)_2 + Cl^- + 4H_2O \longrightarrow Mg_2Cl(OH)_3 \cdot 4H_2O + OH^-$$
(5)



Fig. 12 XRD patterns of corrosion products after shot peening

3.3.2 Electrochemical analysis

Tafel polarization curves of shot peened and untreated samples in 0.9 wt.% NaCl solution are plotted in Fig. 13. The corrosion parameters (e.g. corrosion potential, φ_{corr} and corrosion current density, J_{corr}) obtained from the Tafel polarization curves are listed in Table 4. The results reveal that the HESP treatment, compared with the untreated samples, exhibited excellent corrosion resistance. In all cases, the corrosion potential of the treated samples showed a significant positive shift. Further, samples shot peened for 2, 4, 12 and 15 min had positive shifts of 141, 153, 166 and 203 mV, respectively. Moreover, the corrosion current densities of shot peened WE43 magnesium alloys were reduced by 16.8%, 30.29%, 24.52 % and 31.25%, respectively. This was because that the corrosion resistance of magnesium alloy was determined by the properties of the passivation film $Mg(OH)_2$, which could protect magnesium alloys substrate against being destructed [32]. After HESP treatment, considerable residual compressive stress was generated on the metal surface, thereby increasing the transverse compressive stress of the passivation film and its density. Thus, the corrosion resistance of the substrate could be enhanced. Moreover, residual compressive stress also positively influenced the surface crack initiation and propagation, which could decrease the corrosion rate.



Fig. 13 Tafel polarization curves of WE43 magnesium alloy in 0.9 wt.% NaCl solution

Table 4 Tafel	polarization	curve p	aramaters	of WE43	alloy
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Shot peening time/min	$\varphi_{\rm corr}/{ m V}$	$J_{\rm corr}/({\rm A}\cdot{\rm cm}^{-2})$
0	-1.414	2.08×10^{-4}
2	-1.273	1.73×10^{-4}
4	-1.261	1.45×10^{-4}
12	-1.248	1.57×10^{-4}
15	-1.211	1.43×10^{-4}

4 Conclusions

(1) The mechanical properties were analyzed using microhardness, residual stress, three-point bending and compression tests. The microhardness of WE43 magnesium alloy after shot peening was significantly enhanced, and the maximum increase rate was 110.4%. This also led to the release of surface stress of the sample, resulting in an increase in the residual compressive stress. Besides, the compression resistance and bending resistance were enhanced compared with those of the unpeened bone plate, and the maximum improvement rates were 18.2% and 23.1%, respectively.

(2) The ISNC formation mechanism was that HESP

caused the occurrence of mechanical twinning, which restrained the dislocation movement and led to the increase of a large number of subgrain boundaries. Subsequently, dynamic recrystallization was induced because of the distorted energy, and finally nanograins occurred. Thus, the formation mechanism of the ISCN was achieved through the coordination of twinning, dislocation rearrangement, subgrain boundaries forming and dynamic recrystallization.

(3) HESP generated residual compressive stress on the surface of the sample, which increased the density of the passivation film $Mg(OH)_2$, thereby improving corrosion resistance. In the salt spray corrosion test, the surface corrosion of the shot peening sample was lighter, and the corrosion rate dropped by 29.2% in maximum. Besides, the Tafel polarization curve shows that the corrosion potential of the shot peening samples was significantly shifted in a positive manner, the maximum offset was 203 mV, the corrosion current density dropped by 16%–32%, and the corrosion resistance of the material was better.

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高能喷丸强化镁合金骨板的显微组织、力学和腐蚀性能

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摘 要:为提高镁合金的力学性能和耐腐蚀性能,采用高能喷丸技术(HESP)对其进行强化。结果表明,在镁合金 表面制备原位表面纳米结晶(ISNC)显微组织,其形成机制是通过孪晶、位错、亚晶界形成和动态再结晶相互协调 作用实现的。在样品表面释放应力的作用下,残余压应力和显微硬度增加,从而增加表面钝化膜 Mg(OH)₂的致密 性。此外,腐蚀速率最大降低了 29.2%。在极化曲线中,样品的腐蚀电位最大正向偏移量为 203 mV,腐蚀电流密 度最大下降了 31.25%。另外,骨板的抗压性能和抗弯性能得到改善,最大增加率分别达到 18.2%和 23.1%。因此, HESP 显著改善了镁合金的力学性能和耐腐蚀性能。

关键词: 镁合金; 高能喷丸; 原位表面纳米结晶; 力学性能; 腐蚀性能