Microstructure, Mechanical Properties, Corrosion Behavior and Biocompatibility of As-Extruded Biodegradable Mg–3Sn–1Zn–0.5Mn Alloy

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The microstructure evolution and mechanical properties of biodegradable Mg–3Sn–1Zn–0.5Mn alloys were investigated by the optical microscopy, X-ray diffractometer and a universal material testing machine. The corrosion and degradation behaviors were studied by potentiodynamic polarization method and immersion test in a simulated body fluid (SBF). It was found that the as-extruded Mg–3Sn–1Zn–0.5Mn alloy has the fine equiaxed grains which underwent complete dynamic recrystallization during the hot extrusion process, with the second phase particles of Mg2Sn precipitated on the grain boundaries and inside the grains. The tensile strength and elongation of as-extruded Mg–3Sn–1Zn–0.5Mn alloys were 244 ± 3.7 MPa and 19.3% ± 1.7%, respectively. The potentiodynamic polarization curves in SBF solution indicated the better corrosion resistance of the as-extruded Mg–3Sn–1Zn–0.5Mn alloy in the SBF solution. Immersion test in the SBF solution for 720 h revealed that the corrosion rate of as-extruded Mg–3Sn–1Zn–0.5Mn alloy was nearly 4 ± 0.33 mm/year. The hemolysis rate of as-extruded Mg–3Sn–1Zn–0.5Mn alloy was lower than the safe value of 5% according to ISO 10993-4. As-extruded Mg–3Sn–1Zn–0.5Mn alloy showed good biocompatibility after being implanted into the dorsal muscle and the femoral shaft of the rabbit, and no abnormalities were found after short-term implantation. It was revealed that the as-extruded Mg–3Sn–1Zn–0.5Mn alloy is a promising material for biodegradable implants, which possesses an interesting combination of preferred mechanical properties, better corrosion resistance and biocompatibility.

1. Introduction

In recent years, there has been growing interest in magnesium alloys used as a potential biomaterial in the application of biodegradable implants like stents in vascular medicine or fixation devices for fractured bones in osteosynthesis,[12] due to their interesting combination of mechanical, electrochemical and biological properties. Magnesium degrades naturally in the body fluid and possesses mechanical properties similar to that of natural bone.[3,4] However, magnesium-based implants showed a rapid degradation rate in the physiological environment, which is the main reason for the failure of the implantation before the tissues have healed completely[5,6]. In addition, the relatively low mechanical strength of magnesium is also a limitation to hinder its use in load-bearing applications. Therefore, the strength, elongation and corrosion resistance of magnesium and its alloys need to be improved in order to be used as biodegradable implants.[7–9]. Great efforts have been made in the present study on developing novel magnesium alloys for biodegradable implant applications.

In the present work, quaternary Mg–Sn–Zn–Mn alloy is designed due to the following considerations: (1) Sn: It has been reported that the addition of Sn element is mainly aimed at improving the strength of Mg-based alloys at room temperature[8,10–12]. The solubility of Sn in Mg-based alloys is 14.5 wt%, and mainly functioned as solid solution strengthening element, which forms a Mg5Sn intermetallic phase at a high melting point of 770 °C.
Therefore, Mg–Sn phase could enhance the precipitation strengthening performance of Mg-based alloys due to its morphological and thermal stabilities[13–16]. Regarding the biocompatibility of the alloy, Sn is generally considered as a relatively non-toxic metal. Although Sn and its compounds are poorly absorbed and can accumulate in human tissues, the majority of Sn could be excreted by kidneys rapidly[17]. (2) Zn: The addition of Zn in Mg-based alloys has been reported to improve the mechanical properties and overcome the shortcoming of harmful corrosive effects due to the solid solution strengthening and age hardening[18–21]. In addition, Zn is one of the most abundant nutritionally essential elements in the human body and the released Zn ions can be easily absorbed[22]. (3) Mn: In the case of Mn, it could improve both mechanical properties and corrosion resistance of magnesium alloys, which could form intermetallics and precipitate with other heavy-metal elements[14]. Moreover, Mn plays a primary role in the activation of multiple enzyme system and has no toxic effect except for the extreme occupational exposure[23].

Therefore, Mg–3Sn–1Zn–0.5Mn alloys used in biodegradable implant applications were newly developed in the present work, and the microstructure, mechanical properties, corrosion resistance, hemolysis and animal test of the as-extruded Mg–3Sn–1Zn–0.5Mn alloy had been comprehensively studied.

2. Materials and Methods

2.1. Preparation of specimens and the utilizing solution

The degradable Mg–3Sn–1Zn–0.5Mn alloy in both as-cast and as-extruded state was used in the present study. Mg–3Sn–1Zn–0.5Mn alloy was prepared using purity magnesium (purity not less than 99.70 wt%), pure zinc (purity not less than 99.70 wt%) and Mg–Mn alloy (9.7 wt%) master alloy. Melting was carried out at 750 °C in a high-purity graphite crucible under the protection of a mixed gas atmosphere of SF6, CO2, and Ar. The as-cast ingots of Mg–3Sn–1Zn–0.5Mn alloy were treated with solid solution at 450 °C for 24 h. In the process of extrusion, the solid solution treated alloy was extruded to 370 °C with an extrusion ratio of 16:1. The specimens were polished with silicon carbide sandpapers (grid range of #1000–3000), and then ultrasonically washed in ethanol for 15 min, followed by drying at room temperature.

The standard SBF solution was prepared according to Kokubo’s protocol by dissolving appropriate quantities of the relevant reagent-grade chemicals in deionized water[24]: NaCl, NaHCO3, KCl, K2HPO4·3H2O, MgCl2·6H2O, CaCl2, HCl (1 mol/l), Na2SO4 and NH4Cl (CH3OH). After all the reagents were dissolved, the solution was heated to 37 °C and maintained at this temperature while titrating the solution to a pH of 7.4 with 1 mol/l HCl or NH4Cl(CH3OH)3 using pH meter (Mettler Toledo, FE20). The inorganic ion concentrations in the standard SBF solution are almost the same in human blood plasma (as shown in Table 1).

2.2. Microstructure characterization

The microstructures were observed by optical microscopy (OM, Axiovert 200 MAT). The specimens were prepared by grinding, mechanical polishing and chemical etching in a solution composed of 3% nitric acid for 10 s, and then with 5% picric acid for 10–20 s. The phase structures were detected by X-ray diffraction (XRD, Philips X’Pert PRO).

2.3. Mechanical property evaluation

Tensile tests were carried out on a universal material testing machine (Instron 3365, USA) at a constant crosshead speed of 0.5 mm/min at room temperature. Three specimens were tested for alloy.

2.4. Immersion test

The immersion test was carried out in water bath at 37 ± 0.5 °C. Each specimen was put into a tightly closed tube and the ratio of solution volume to specimen surface is 25 mL/cm2 according to ASTM G31–72[23]. The specimens were immersed for 30 days and the corrosion rate \( P_w \) (mm/year) was calculated according to formula (1)[25,26], in which \( W_0 \) is the dry weight of each specimen before immersion test, \( W_i \) is the dry specimen weight after the immersion test and the corrosion products and \( \rho \) is the metal density. An average of three measurements was taken for each group.

\[
P_w = \frac{87.6(W_0 - W_i)/\rho}{t}
\]

After immersing for 96 h, 240 h and 720 h in SBF, the specimens were fully rinsed in double distilled water and dried subsequently. The corroded surfaces were monitored by scanning electron microscopy (SEM, Quanta 200). Elemental mappings from the corroded surfaces were acquired by using energy dispersive X-ray spectrometry (EDS). The phase constituents in the corrosion products were determined by X-ray diffraction (XRD; Philips X’Pert PRO, CuKα irradiation).

2.5. Potentiodynamic polarization test

The electrochemical experiments were conducted in SBF at 37 °C on an electrochemical workstation (CHI660C, Shanghai Chenhua Co., China) using a three-electrode system with the specimen as the working electrode, Pt sheet as the measuring electrode, and calomel electrode as reference electrode. Once contacting with the test solution, severe corrosion took place. Therefore, the potential scanning started immediately as soon as the specimen was exposed to the test solutions. The electrolyte volume was 200 mL and the exposure area was 0.283 cm2 for the test specimens. The scanning potential range was from –2.0 to –0.8 V and the potential scanning rate was 1 mV/s in the polarization test.

2.6. Hemolysis tests

Hemolysis tests were conducted according to the instruction of ISO 10993–4[24]. Healthy human blood anticoagulated by heparin sodium was drawn and diluted with normal saline at a volume ratio of 4:5. The specimens were separately dipped in a standard tube containing 10 mL of normal saline and then incubated at 37 °C for 30 min. Then 0.2 mL of diluted blood was added to the standard tube and incubated at 37 °C for another 60 min. Normal saline solution and ultrapure water were used as negative and positive controls, respectively. Afterward, all the specimens were removed and all the tubes were centrifuged at 3000 r/min for 5 min. Then the optical density (OD) of each supernatant was measured using an ultraviolet and visible spectrophotometer (UV-Vis, Agilent 8453, USA) at 545 nm. Three replicates were conducted for each alloy. The hemolysis percentage was calculated by Eq. (2).
Hemolysis = \left[ \frac{\text{Absorbency (test)} - \text{Absorbency (negative control)}}{\text{Absorbency (positive control)} - \text{Absorbency (negative control)}} \right] \times 100\% \quad (2)

2.7. Animal testing

In vivo the animal testing was performed according to the Hospital of Heilongjiang Province. The implants were machined in rod shape with 2 mm in diameter and 4 mm in length. Adult rabbits (provided by the Animal Experiment Center of the 2nd affiliated Hospital of Harbin Medical University) with 2.0–2.5 kg weight were anesthetized with 10 mg/kg Zoletil, and 10 μg/kg L DEX pyrimidine. Adult rabbits were randomly divided into 3 groups with two rabbits in each group (marked group A and group B for the test samples and a control group). Mg alloy samples were implanted in the dorsal muscle in group A and in the femoral shaft in group B, as shown in Fig. 1.

The serum magnesium in the blood (UniCelDxC 800, Beckman Coulter Co. Ltd.) was taken from marginal ear vein of the rabbits before experiment and at different implantation time points. The X-ray radiography (MIKASA HF100H, MIKASA X-RAY Co. Ltd) was carried out on the implanted area at different time points for observation of the implant degradation. Rabbits were sacrificed at the planned time points using 0.3% sodium pentobarbital solution for overdose anesthesia, and the organ tissue samples of the heart, liver, kidney and spleen were obtained. Pathological examination with HE staining (hematoxylin–eosin staining) was conducted for observation of any pathological changes around the implant.

3. Results and Discussion

3.1. Microstructure

Fig. 2 shows optical micrographs of Mg–3Sn–1Zn–0.5Mn alloys in both as-cast state and as-extruded state. It could be observed that the microstructure of as-extruded Mg–3Sn–1Zn–0.5Mn alloy was fine equiaxed grains, while the as-cast Mg–3Sn–1Zn–0.5Mn alloy showed inhomogeneous microstructure with α-Mg dendrites grains. The grain size of the as-cast Mg–3Sn–1Zn–0.5Mn alloy was bigger than that of as-extruded alloy. The morphology of α-Mg dendrite grains of as-cast Mg–3Sn–1Zn–0.5Mn alloy was similar to that of
the previous report\cite{29}, which was ascribed to the partition of Sn in the liquid ahead of the solidification front, resulting in a constitutional supercooling zone of liquid ahead of the interface. On the other hand, fine equiaxed grains of as-extruded Mg–3Sn–1Zn–0.5Mn alloy indicated that it underwent complete dynamic recrystallization during the hot extrusion process\cite{30} accompanied with the large deformation and high deformation temperature. The second phase particles of Mg$_2$Sn in the form of small dark dots on the grain boundaries and inside the grains of α-Mg for the as-extruded alloy could also be seen in Fig. 2(b).

Fig. 3 presents the X-ray diffraction patterns of as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy, from which the expected matrix phase α-Mg and second phase Mg$_2$Sn can be clearly distinguished. The intensity of Mg$_2$Sn phase diffraction peak was higher for the as-extruded alloy in comparison with the as-cast counterpart.

3.2. Mechanical property

The mechanical properties of as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy samples are presented in Fig. 4. The tensile strength and elongation of as-extruded Mg–3Sn–1Zn–0.5Mn alloy (244 ± 3.7 MPa, 19.6% ± 1.7%) were higher than those of as-cast ones (156 ± 5.6 MPa, 14.7% ± 1.5%), indicating better mechanical properties of the as-extruded Mg–3Sn–1Zn–0.5Mn alloy. This can be mainly attributed to the grain refinement due to the dynamic recrystallization during the hot extrusion process and the increased volume fraction of Mg$_2$Sn particles after extrusion\cite{31}, as indicated in Figs. 2 and 3. The Mg$_2$Sn phases played an important role in the deformation behavior of Mg and its alloys at room temperature, which is possibly related to the following two aspects: (1) the presence of fine and uniform phases distributed along the grain boundaries easily acts as an effective straddle to the dislocation motion thus improving the properties of alloy\cite{31}; (2) the presence of second phase particles may also act as crack sources which is detrimental to ductility\cite{32,33}.

3.3. Potentiodynamic polarization test

Potentiodynamic polarization test in SBF solution was carried out to evaluate the biodegradation behavior of as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy (Fig. 5). Table 2 exhibits the parameters including the corrosion current density ($I_{corr}$), corrosion potential ($E_{corr}$) and the average corrosion rate ($V_{corr}$) obtained from the potentiodynamic polarization curves. It could be seen that the corrosion current density of Mg–3Sn–1Zn–0.5Mn alloy in SBF solution increased in the as-extruded state compared to that in the as-cast state, indicating better corrosion resistance of as extruded Mg–3Sn–1Zn–0.5Mn alloy. The correlated average corrosion rate in Table 2 confirmed this trend. Many investigations revealed that the microstructure, such as grain size and second phase, affected the corrosion rate of magnesium alloys\cite{33–37}. On the one hand, the reduction of grain size led to the increased corrosion resistance of magnesium alloys\cite{34,37}. On the other hand, the increase of second phase resulted in the decreased corrosion resistance of magnesium alloys, probably due to the enhanced microgalvanic corrosion\cite{34}. In the present study, the microstructure of as-extruded alloy was fine equiaxed grains and the amount of second phase of as-extruded

<table>
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<tr>
<th>Status</th>
<th>$E_{corr}$ (V)</th>
<th>$I_{corr}$ (μA/cm$^2$)</th>
<th>$V_{corr}$ (mm/year)</th>
</tr>
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<tbody>
<tr>
<td>As-cast</td>
<td>−1.78</td>
<td>25.12</td>
<td>5.66</td>
</tr>
<tr>
<td>As-extruded</td>
<td>−1.68</td>
<td>13.18</td>
<td>2.97</td>
</tr>
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</table>

Fig. 4. Mechanical properties of as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy.

Fig. 5. Potentiodynamic polarization curves of as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF solution at 37°C.
alloy was not significantly increased than that of the as-cast one. Therefore, the as-extruded alloy showed better corrosion resistance.

3.4. Hemolysis

Hemolysis ratio is used to assess the destructive effect of a medical material to the erythrocyte. Fig. 6 shows the hemolysis results of as-extruded Mg–3Sn–1Zn–0.5Mn alloy. As shown in Fig. 6, the hemolysis ratios of Mg–3Sn–1Zn–0.5Mn alloy were 6.8% ± 0.9% and 4.7% ± 0.4% for the as-cast and as-extruded alloy, respectively. According to the ISO 10993-4[28], the hemolysis ratio of implant materials to be used in medical applications has to be less than 5%. Moreover, the hemolysis ratio of as-extruded Mg–3Sn–1Zn–0.5Mn alloy was within the judging criterion now.

3.5. Immersion test

The variation of the pH value of as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF solution as a function of immersion durations during the immersion test is shown in Fig. 7. It could be found that the pH value of as-extruded Mg–3Sn–1Zn–0.5Mn alloy solution increased with increasing immersion time. The pH value increased rapidly in the initial 10 h. After 144 h immersion, the pH values of as-extruded Mg–3Sn–1Zn–0.5Mn alloy increased from 7.4 ± 0.02 to nearly 10 ± 0.06. Afterward, the pH value of SBF increased slowly with increasing immersion time and became stabilized at 10.2 ± 0.03.

When Mg alloys were immersed in SBF solution, the degradation occurred in SBF solution according to the following reaction:

\[ \text{Mg} + \text{H}^+ + \text{H}_2\text{O} \rightarrow \text{Mg}^{2+} + \text{OH}^- + \text{H}_2 \]  

(3)

Consequently, in the early stage of immersion, the dissolution of magnesium consumed H\(^+\), but released OH\(^-\), leading to the increase of pH value of SBF[38]. Subsequently the corrosion rate of the specimens decreased noticeably due to the formation of Mg(OH)\(_2–\)nH\(_2\)O protective layer. Through the formation of this protective layer, the CO\(_3^{2-}\), PO\(_4^{3-}\) and Cl\(^-\) ions were attracted to the surface of the specimen, causing more accumulation of OH\(^-\) ion that is vital for apatite nucleation[39–41].

Fig. 8 shows the corrosion rate of as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF solution for different duration time. The weight loss of magnesium alloy was an indication of corrosion[42]. It could be seen in Fig. 8 that the corrosion rate of as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF solution reduced with increasing immersion. The corrosion rate dropped rapidly in the initial 120 h and then slowly with increasing immersion time. The corrosion rate of the solution corresponding to as-extruded Mg–3Sn–1Zn–0.5Mn alloy became stabilized at nearly 4 mm/year at the end of the immersion test. The initial degradation of magnesium resulted in the rapid formation of corrosion product layer, Mg(OH)\(_2\). However, due to the presence of aggressive chloride ions and the non-compact characteristics of the protective layer, corrosion occurred rapidly because of the formation of massive corrosion products such as MgO/Mg(OH)\(_2\), magnesium/calcium phosphates and carbonates and so on. After immersion durations, formation of corrosion products reduced active regions and suppressed further dissolution of the substrate.

Surface morphologies of as-extruded Mg–3Sn–1Zn–0.5Mn alloy after immersion in SBF solution for different time are shown in Fig. 9(a, c and e). As can be seen from Fig. 9(a), a number of cracks appeared on the surface of as-extruded Mg–3Sn–1Zn–0.5Mn specimen for 96 h, and the surface of the specimen could be seen with obvious corrosion products over the cracks boundaries. The enlarged views of local area outside the obvious corrosion products
showed that the surface presented a crackled appearance due to the dehydration of the corrosion film after drying in cold air. The presence of Cl$^-$ in the solution promoted the corrosion, because the corrosive intermediate (Cl$^-$) was rapidly transferred through the outer layer and reached the substrate of the alloy surface. Hence, the corrosion was increased. It could be seen in Fig. 9(c) that a great number of irregular shallow pits presented on the surface of Mg–3Sn–1Zn–0.5Mn alloy specimen after immersion for 120 h, which may be caused by the flaking off of the second phase Mg$_2$Sn from the surface of specimen when the as-extruded Mg–3Sn–1Zn–0.5Mn alloy suffered the severest attack. After immersion for 240 h, plenty of deep and large corrosion pits with rough bottom could
Fig. 10. XRD patterns of as-extruded Mg–3Sn–1Zn–0.5Mn alloy after immersion in SBF solution for different time.

be observed on the surface of Mg–3Sn–1Zn–0.5Mn alloy specimens, as well as the flecked-off surface feature and the disintegrated fragments, as shown in Fig. 9(e). Fig. 9(b, d and f) shows the EDS analysis results of as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF for different immersion times, indicating the presence of oxygen, carbon, magnesium, calcium and phosphorus elements, which was in agreement with the recent research [44,45].

The XRD patterns of Mg–3Sn–1Zn–0.5Mn alloy after immersion in SBF solution for different time durations are shown in Fig. 10. The diffraction peaks corresponding to α-Mg phase and Mg6Sn phase were found in the XRD patterns for 96 h, probably due to the low volume fractions of the second phase. On the other hand, Mg(OH)2 peaks could be clearly identified for the specimens after immersion for 240 h and 720 h. In addition, the diffraction intensities of Mg(OH)2 phases increased with increasing immersion durations.

The presence of Mg(OH)2, carbonates and phosphate was also supported by the EDS results (Fig. 9), which revealed the presence of oxygen, carbon, magnesium, calcium and phosphorus elements. Therefore, the corrosion product layer on the surface of as-extruded Mg–3Sn–1Zn–0.5Mn alloy after immersion in SBF solution consisted of magnesium (calcium) phosphates, magnesium (calcium) carbonates and magnesium hydroxide, which was consistent with the results reported in literature [46].

3.6 Animal implant experiments

Magnesium ion concentration in the serum fluctuates in the range of 1.15–1.75 mmol/l [47], while the normal range of serum magnesium concentration in experimental animals is 0.82–2.22 mmol/l. To detect the changes of serum magnesium level in the blood plasma for as-extruded Mg–3Sn–1Zn–0.5Mn alloy before implantation and after different periods of post-operation, the concentrations of magnesium ions in the serum of rabbit blood were measured by blood test, as shown in Table 3. After surgery, the serum magnesium concentrations increased due to the corrosion of magnesium substrate in the physiological environment, which was within the normal range of physiological magnesium level. The degradation of as-extruded Mg–3Sn–1Zn–0.5Mn alloy implants had little effect on the blood concentration of magnesium ions, which indicated that the liver and kidney functions of the experimental animals were in good condition so that the excess Mg2+ was excreted in experimental animals. Hartwig [48] believes that the intake of a high concentration of magnesium ions would not cause adverse reactions; powerful excretory system of kidney and storage buffer function of bones can make the body maintain the balance of serum magnesium concentration.

Fig. 11 shows X-ray radiography images from subcutaneous implants (Fig. 11(a, c, e, g, i and k)) and orthopedics implants (Fig. 11(b, d, f, h, j and l)) of as-extruded Mg–3Sn–1Zn–0.5Mn alloy at different time points after surgery. The gas bubbles around the implants (black area) were observed in Fig. 11 at different time points after surgery, which was ascribed to the generated hydrogen gas derived from the reaction of the implanted magnesium alloy with the body fluid. The gas accumulated in the fibrous capsule and formed a visible air mass because of the hysteresis and limitedness of metabolism, thus the amount of gas formed in per unit time was so large than beyond the ability of the body to normally metabolize. It is worth noting that the absorption of hydrogen gas usually occurred in longer time as a result of decreasing the corrosion rate due to the formation of corrosion products on the surface [49]. Moreover, it has been reported that an appropriate release rate of hydrogen would not cause damage to the body, and on the contrary, it could play a role in eliminating free radical and inflammation [50].

As can be seen in Fig. 11, magnesium alloy rod implanted into different positions showed different degradation rates. The degradation rate of the as-extruded Mg–3Sn–1Zn–0.5Mn alloy rod implanted in dorsal muscle was relatively lower than that implanted in the femoral shaft. It was considered that the different degradation behaviors were derived from the different implantation positions in the body. The slower degradation rate of the alloy rod was ascribed to the wrapping by the tissue when the material was implanted into the dorsal muscle. On the other hand, when implanted into the femoral shaft, the degradation rate was greater because most of the alloy rod was located in the bone marrow cavity and it suffered from scouring by body fluid.

Fig. 12 shows the histological images of rabbit abdominal organs and tissues after being implanted for 6 months of as-extruded Mg–3Sn–1Zn–0.5Mn alloy samples, including heart (Fig. 12(a and f)), liver (Fig. 12(b and g)), kidneys (Fig. 12(c and h)), spleen (Fig. 12(d and i)), and tissue around implants (Fig. 12(e and j)). According to the histological images, no inflammation and pathological alteration was observed in all organs and tissues around the implants in the present experiment, indicating good biocompatibility of

<table>
<thead>
<tr>
<th>Research groups</th>
<th>Mg2+ concentration in the serum (mmol/l)</th>
</tr>
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<tbody>
<tr>
<td>Level of Mg2+ concentration in serum (mmol/l)</td>
<td>1</td>
</tr>
<tr>
<td>Before implants</td>
<td>1.3 ± 0.2</td>
</tr>
<tr>
<td>After implantation, time (d)</td>
<td>1.26 ± 0.14</td>
</tr>
</tbody>
</table>

Table 3

Mg2+ concentrations in the serum of rabbit blood for as-extruded Mg–3Sn–1Zn–0.5Mn alloy samples before and after implantation.
as-extruded Mg–3Sn–1Zn–0.5Mn alloy with animal organs (heart, liver, kidney and spleen) and tissues. It can be inferred that the as-extruded Mg–3Sn–1Zn–0.5Mn alloy implanted in animals at the early stage was biosafe and the experimental animals tolerated the degradation products of as-extruded Mg–3Sn–1Zn–0.5Mn alloy.

4. Conclusion

In the present study, a novel Mg–3Sn–1Zn–0.5Mn alloy with excellent mechanical properties and biocompatibility for biodegradable applications was developed through alloy designing. Besides, extrusion could be additionally performed on this alloy to get better performance. The results showed that both as-cast and as-extruded Mg–3Sn–1Zn–0.5Mn alloy were composed of α-Mg and Mg5Sn phases, and the as-extruded Mg–3Sn–1Zn–0.5Mn alloy contained fine equiaxed grains due to the complete dynamic recrystallization while as-cast Mg–3Sn–1Zn–0.5Mn alloy possessed inhomogeneous microstructure of α-Mg dendrites grain. In addition, the mechanical properties, corrosion resistance and hemolysis of as-extruded Mg–3Sn–1Zn–0.5Mn alloy were superior to those of as-cast alloy. The immersion test of as-extruded Mg–3Sn–1Zn–0.5Mn alloy in SBF solution for different durations showed good corrosion properties, whose pH value and corrosion rate became stabilized at nearly 10 and 4 mm/year during the long time immersion, respectively. The hemolysis rate of as-extruded Mg–3Sn–1Zn–0.5Mn alloy was 4.7%, which was in the acceptable range of human body (5%). The results of animal test indicated that the magnesium concentration in the serum of as-extruded Mg–3Sn–1Zn–0.5Mn alloy fluctuated in the normal range, and there were no significant histological changes of the various organs and tissues. Based on the good mechanical properties, corrosion properties and blood biocompatibility, the as-extruded Mg–3Sn–1Zn–0.5Mn alloy showed promising prospects as biodegradable implant material.

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Fig. 11. X-ray images of as-extruded Mg–3Sn–1Zn–0.5Mn alloy samples implanted in rabbits.

Fig. 12. Histological images of rabbit abdominal organs and tissues after implantation of as-extruded Mg–3Sn–1Zn–0.5Mn alloy samples for 6 months.
References