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Manufacture of biodegradable magnesium alloy by high speed friction stir processing

Dingqiang Qin[a], Haorui Shen[a], Zhikang Shen[a,c], Haiyan Chen[a,b,c], Li Fu[a,b,c,*]

[a] School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an, 710072, China
[b] State Key Laboratory of Solidification, Xi'an, 710072, China
[c] Shaanxi Key Laboratory of Friction Welding Technologies, Xi'an, 710072, China

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ABSTRACT

Friction stir processing (FSP) with a high rotational speed (6000 rpm) was employed to modify surface properties of magnesium alloy through grain refinement and incorporated nano-hydroxyapatite (nHA) particles. Hardness testing indicated that the distributed nanoparticles have largely influenced the hardness of the surface layer and measurements of average hardness varied with different amounts of nHA particles applied. Corrosion tests revealed that FSP had reduced the corrosion rate of ZK60 magnesium alloy by more than one order of magnitude due to the combined effects of grain refinement and low surface energy. The corrosion rates of modified layers could be manipulated by nHA content of selection since nHA plays an important role in destroying the continuity of magnesium matrix and producing a more protective biomineralized film on composites when immersed in simulated body fluid (SBF). Two kinds of equivalent circuits were proposed to explain the corrosion mechanism. The simulation values fitted the experimental data very well by using the equivalent circuits, regardless of differences in samples.

1. Introduction

Over the last few years, magnesium and its alloys have become intriguing biomaterials to many researchers due to their properties of non-toxicity, desirable biodegradability and biocompatibility [1]. In comparison to the conventional metallic materials, Young's moduli of magnesium and its alloys show great similarity to natural bone, which in turns could effectively minimize the effects of stress shielding. The greatest advantage of magnesium alloy implants is that it can be fully degraded after bone tissue healing and the removal of the material by a second surgery procedure is not required, which is mandatory to the conventional metallic materials [1,2]. However, the rapid corrosion rate of magnesium has been reported by previous studies, especially in the early stage of degradation phase [3]. Surface degradation with the formation of surface alkalization and hydrogen foams is able to affect the tissue growth [4]. Therefore, the modification on the surface of magnesium alloys is generally required to assist in the prevention of fast corrosion in the initial stage of the degradation phase.

Surface modification can be achieved by a number of commonly-used techniques, including surface coating [5–7], ion implantation [8–10], and laser surface melting [11,12]. These methods are difficult to avoid the metallurgical defects formed by the strong chemical reactions between the reinforcements and matrix. Thus, Friction stir processing (FSP) has been developed to modify surface microstructure or produce metal matrix composites (MMCs), which are composed of aluminium, copper, magnesium and titanium [13]. Powder metallurgy, laser-assisted process and plasma spraying are the commonly used techniques for fabrication MMCs. During these processes, materials are transformed from a solid phase to a liquid state due to the effect of high temperature, and adverse reactions took place between base metal and additives. Therefore, FSP, as a solid-state processing technique, is highly recommended to use since it could avoid the potential disadvantages of using the conventional liquid phase processing. A rotating tool with a specially designed pin and shoulder is plunged into a plate and then traverses along the scheduled line accompanied with severe plastic deformation and localized heating. As a result, the formation of grain refinement via dynamic recrystallization can be easily achieved [14].

Hydroxyapatite (HA), a natural mineral compound of calcium apatite and a major component of natural bone, attracts great interests to researches as a promising bioceramic material in the past two decades. Hahn et al. [15] manufactured a compact and well-appendiculate HA-chitosan composite coating on the AZ31 magnesium alloy. The coated alloys were found to have a better corrosion resistance than the

* Corresponding author at: School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an, 710072, China.
E-mail address: fuli@nwpu.edu.cn (L. Fu).

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magnesium alloys alone, and its biocompatibility was obviously improved. Although the HA coatings have many advantages, they appear to be brittle and can be fragile in the load-bearing conditions [6]. Furthermore, bonding between the coatings and substrates would be significantly affected when electrolyte permeates the coatings through a large crevice and corrodes the magnesium substrates. Fabricating MMCs is an alternative beneficial way described in many literature studies because of their compatibility in an application with different additives. Substances containing bioactive elements such as Ca and P are widely used as reinforcements for controlling the corrosion rate and enhancing the biocompatibility of magnesium alloys. In the study conducted by Witte et al. [16], HA particles in the cell solutions and artificial seawater enhanced the corrosion resistance of magnesium matrix. Reduction of the material degradation has been reported by He et al. [17] due to adding pearl powders and showed improvements on adhesion and proliferation of the bone cells. In the study investigated by Sunil et al. [18], which HA particles reinforced AZ31 magnesium matrix was produced by FSP, the effect of HA particles on the corrosion behaviour of FSPed composites was not well-studied initially, and concern was raised on AZ31 matrix contains element Al due to their substantial toxicologically [19]. Most studies about fabricating MMCs by FSP employed a relatively lower rotational speed between 1000 rpm and 2000 rpm, which could have possibly resulted in an uneven distribution of powders over the substrate and influenced the properties of MMCs. In this study, ZK60 magnesium containing non-toxic elements of Zn and Zr as base metal was used [20]. Fabricated ZK60-nHA composite was produced using FSP with a high rotational speed of 6000 rpm. The objective of the study was to investigate the effect of FSP rotational speed on structural modification or the impact of high rotational speed process on structural modification and the influence of nHA content on the corrosion behaviour of ZK60 magnesium matrices.

2. Experimental methodology

2.1. Materials and processing

The size of 120 × 120 × 4 mm^3 ZK60 cast magnesium alloy sheet was prepared, whose composition is Mg bal., 4.8 ~ 6.2 Zn, 0.3 ~ 0.9 Zr, 0.1 Mn, 0.05 Al (wt.%). The FSP tool was produced by hardened H-13 tool steel with an 8 mm diameter triple spiral shoulder and a cone-shaped pin. The diameter of pin varied from 3.5 mm at the root to 2 mm at the top, and the pin was 1.7 mm in length. For the 4 mm thick plate, the length of the pin was 1.7 mm, which was to avoid changing the original characteristics of the magnesium alloy in a large part, and it was ensured that the obtained samples can retain the substrate properties of non-toxic, desirable biodegradability and biocompatibility of the magnesium alloys as much as possible. In order to achieve defect-free samples, the processing parameters were optimized by varying the four rotating speeds of 3000 rpm, 4000 rpm, 5000 rpm and 6000 rpm with a constant processing speed of 100 mm/min. The FSP was performed on the FSW-TS-F08-DZ machine. The process of friction stir processing is shown in Fig. 1. Several parallel grooves with 0.35 mm width and 1.2 mm depth were produced regular interval on the surface of a sheet. After placing the nHA powders with an average size of about 100 nm into the grooves, a pin-less tool closed the upper space of the grooves in order to prevent the powders spreading out during the FSP (Fig. 1(a) and (b)). The morphology of the nHA powders is shown in Fig. 2. The pin-less tool was produced by hardened H-13 tool steel with a 6 mm diameter triple spiral shoulder. It employed to cover the grooves by the constant processing parameters of 5000 rpm and 300 mm/min. Then, the tool with a pin traversed along the grooves one at a time from the first groove to the last (the first pass) (Fig. 1(c)) and subsequently it returned to the first groove along the way opposite to the former route (the second pass) (Fig. 1(d)). For the calculation of nHA weight fraction, the quantity of applied nHA particles was measured by weighing the sheet before and after placement of nHA to the grooves. The cross-sectional area of stir zone was determined by analyzing the macro-graphic image. By varying intervals of the parallel grooves, based on the volume size of stir zone (SZ), mass of incorporated nHA and density of nHA and ZK60 or while maintaining conditions of the volume size of SZ, mass of incorporated nHA and density of nHA and ZK60, the fabrication of ZK60-nHA composites was produced with different weight percent of nHA.

2.2. Microstructural characterization

Samples for the characterization of microstructure were cross-sectioned perpendicular to the processing direction. They were ground with SiC papers to 3000 grade and polished with a diamond paste (0.5 μm) as per standard metallographic procedure. Oxalic acid reagent (1 g oxalic acid and 1 ml nitric acid were dissolved in 150 ml water for 4–6 s) was applied to etch the samples for the microstructure study. Optical microscope (OM, OLYMPUS PMG3, Japan) and scanning electron microscopy (SEM, SUPRA 55, ZEISS, German) equipped with an X-ray energy dispersive spectroscopy (EDS, Oxford INCA) were used to identify the composition and structure of the corroded surfaces. The specimens were then characterized by X-ray diffraction analysis equipment (X' Pert PRo MPD, PANalytical).

2.3. Hardness testing

The hardness of magnesium alloys and prepared nanocomposites were evaluated by using HMV-1 Vickers microhardness tester and its technique (Leitz, Germany) under an applied load of 0.98 N for 15 s. The measured values were obtained along a centreline across the samples' transversal surface at a constant interval of 0.5 mm, with hardness distribution and average values were reported.

2.4. Electrochemical measurement

Simulated body fluid (SBF) was used to investigate the corrosion behaviour of the assessed specimens. The reagents required to prepare SBF are shown in Table 1 [21]. The electrochemical testing was carried out in SBF at a temperature of 37 °C and buffered at PH = 7.4. A three-electrode electrochemical cell composed of a broad platinum network as a counter-electrode, a reference electrode of Ag/AgCl and an operational electrode of the samples was selected. All specimen surfaces were ground to 1500 grade by using SiC sandpapers. The area of assessed specimens immersed in the SBF was 1 cm^2. The measured open circuit potential (OCP) was a function of the time, and the OCP of the sample in the SBF was recorded rapidly. The electrochemical impedance spectroscopy (EIS) was performed between 0.01 Hz and 100 MHz under the OCP. In order to achieve a stable behaviour state, the initial delay and sinusoidal wave were 20 min and 10 mV, respectively. The testing data obtained from the EIS was analyzed by using the USA commercial software Zsimpwin. Potentiodynamic polarization study was carried out at a 2 $mV \cdot s^{-1}$scanning speed and a 20 min initial delay, to achieve a stable state for equipment for obtaining consistent data measurement.

3. Results and discussion

3.1. Processing parameter optimization

The distribution of nHA powders in the base metal with varying processing parameters is shown in Fig. 3. In most studies, researchers employed FSP with a relatively low tool rotational and processing speed to fabricate magnesium matrix composites [22,23]. As a result, several banded structure aggregations of additives and minute crevices probably appeared in the base metal because of the inadequate heat input as well as the insufficient material flow. In order to investigate the effect of rotational speed on the distribution of nHA, the rotational speed was

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