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Research paper

Effects of backward extrusion on mechanical and degradation properties of Mg–Zn biomaterial

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ARTICLE INFO

Article history:
Received 4 December 2011
Received in revised form
24 February 2012
Accepted 27 February 2012
Published online 4 March 2012

Keywords:
Magnesium alloys
Biomaterial
Microstructure
Mechanical properties
Corrosion properties

ABSTRACT

Backward extrusion was used to improve the properties of Mg-based biomaterials. The microstructures, mechanical performance and corrosion properties of as-cast and backward extruded Mg–xZn (x = 0.5, 1, 1.5, 2, wt.%) alloys were investigated. The secondary dendrite arm spacing of as-cast Mg–xZn alloys and the grain size of backward extruded Mg–xZn alloys were decreased with the increment of Zn content. Meanwhile, both strength and elongation were improved by backward extruded treatment. With increasing Zn addition, the corrosion properties of both as-cast and backward extruded Mg–xZn alloys were decreased. However, the corrosion performance of backward extruded sample was improved obviously compared to the corresponding as-cast one. More importantly, the degradation rate of the backward extruded alloy was stable, which was mainly associated with the fine second precipitates and the homogeneous microstructure. It was demonstrated that backward extrusion was an effective approach to manufacture high performance Mg-based biomaterials.

1. Introduction

Magnesium (Mg) alloys have received increasing interest due to their potential applications as a new generation of biomedical implants. At the early stage of Mg biomaterials, some commercial Mg alloys used as engineering materials were adopted to study as biomaterials directly, such as AZ91, AZ31, AM60B, WE43 etc. (Gu et al., 2010; Witte et al., 2008, 2010; Zeng et al., 2008). The strength, creep properties and thermal stability are the main concerns during the optimizing processes (Beals et al., 2007; Ganeshan et al., 2009; Luo, 2002; Song, 2005). The issue of biocompatibility has rarely been considered. The harmful effects of the widely used elements in structural Mg alloys, such as aluminum (Al) (Shaw and Petrik, 2009), manganese (Mn) (Acharya and Park, 2006), zirconium (Zr) (Ghosh et al., 1990) and rare earth elements (Ferreira et al., 2003; Feyerabend et al., 2010) on human health have been confirmed. As a result, the potential bio-toxicity of degradation products has been the inevitable problem for the applications of commercial Mg alloys as orthopedic biomaterials.

Recently, the guideline of the alloying elements that existing in the human body has been proposed to minimize the cytotoxicity. Based on this guideline, a series of new alloys have been developed. It has been reported that hydrogen

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doi:10.1016/j.jmbbm.2012.02.024

evolution has rarely observed during the corrosion process in MgZnCa glass implants, which provided a new method to develop new biodegradable implants (Zberg et al., 2009). Moreover, it was found that the Mg–6 wt%Zn alloy exhibited suitable mechanical strength, good biocompatibility and moderate degradation rate (Zhang et al., 2010). In addition, the effects of Ca element on the bio-corrosion behavior and the cyto-compatibility of Mg have been studied, and it was suggested that the Mg–1 wt% Ca alloy was one of the promising orthopedic biodegradable materials (Gu et al., 2011). More attractively, the binary Mg–RE (RE: Y, Gd and Dy) alloys were also introduced as the novel biomaterials for the fine implants or degradation stents (Hort et al., 2010). At present, the development of Mg-based biomaterials mainly concentrated on the alloy development via alloying effects. Limited attention has been paid on the issues of preparing technology, which directly influenced the following mechanical and corrosion properties. For instance, most of these developed alloys were prepared by gravity cast or deformation, whereas there existed a plenty of defects in as-cast state Mg alloys, which straightly affected the mechanical integrity during the decomposed process (Friedrich and Mordike, 2006). The rolling and forward extrusion technologies were performed to improve the mechanical properties of Mg-based alloys (Hou et al., 2009; Liu et al., 2009). Unfortunately, a hardened layer tends to form on the surface of the sample. The grain size, especially in the middle of the sample, was larger than that on the edge due to the different strains during the deformation. Thereby, the mechanical properties vary evidently from the edge to the center, leading to low formability of stent.

Zn is an essential trace element for human beings, but it is critical for biological functions (Berman, 1980). Therefore, the Mg–Zn binary alloy is an ideal biomaterial. However, the low formability and irregular decomposition rate are still the shortcomings of Mg–Zn biomaterial. In this work, backward extrusion was performed to improve the deformability and degradation properties of Mg–Zn biomaterial. The microstructures, mechanical properties and degradation properties of as-cast (AC) and backward extruded (BE) Mg–Zn alloys were investigated.

2. Materials and methods

2.1. *Preparation of materials*

Mg–xZn (x = 0.5, 1, 1.5, 2; wt.%) alloys have been prepared by zone solidification technology. The zone solidification method was characterized that the ingot was gradually cooled by water. This method was specially developed to prepare high pure Mg based biomaterials. The detailed description and purifying mechanism have been reported in our previous literature (Peng et al., 2010). The alloys have been prepared in a steel crucible under protection by a mixture of CO_2 and SF_6. After mixing at 720 °C for 1 h, the alloy was cast to a mold preheated at 600 °C. The filled mold was held at 670 °C for 1 h under the protective gas. Then, the whole steel crucible with the alloy was immersed into the cold water at 30 mm/s. When the bottom of the steel crucible touched the water, it stopped for 2 s. As soon as the liquid level of the crucible inside was aligned with the height of water outside, the solidification process was finished. The chemical composition of the alloys was measured by X-ray fluorescence spectroscopy. The detailed composition is listed in Table 1. It can be seen that the content of the alloying element was lower than that of nominal composition owing to the burning loss. The main impurities are lower than 0.015 wt%.

Table 1 – The detailed chemical composition of the alloys.

Alloys	Elements (wt.%)				
	Zn	Fe	Ni	Cu	Mg
Mg–0.5Zn	0.92	0.004	0.001	0.004	Bal.
Mg–1.0Zn	0.91	0.008	0.005	0.004	Bal.
Mg–1.5Zn	0.96	0.007	0.004	0.006	Bal.
Mg–2.0Zn	0.93	0.004	0.003	0.007	Bal.

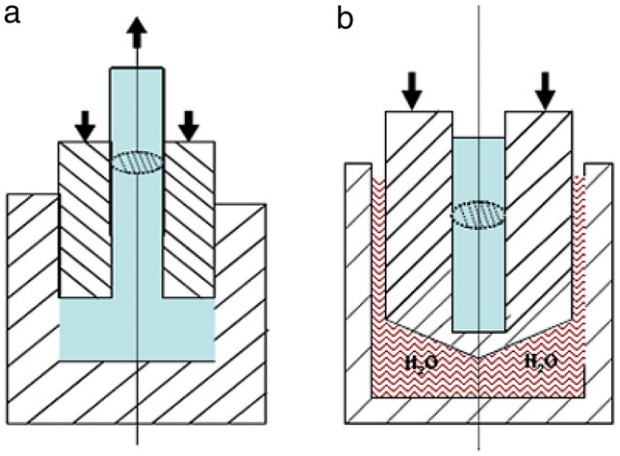

Fig. 1 – Schematic representation: (a) BE technology; (b) the zone solidification method (Peng et al., 2010).

The sample of 70 mm in diameter was heat treated. The solution treatment was performed at 530 °C for 10 h, and then it was quenched in hot water (60 °C). The backward extruded temperature was 420 °C. The ingot was preheated at 420 °C for 1 h and then it was backward extruded immediately. The extruded ratio and speed were 12.25 and 50 mm/min, respectively. The schematic representation was shown in Fig. 1(a). By comparison, the same size as-cast state samples were prepared by the zone solidification method (Fig. 1(b)).

2.2. *Microstructure characterization*

The microstructural investigations were performed using optical microscopy (OM) and transmission electron microscopy (TEM) equipped with energy dispersive X-ray analysis (EDX). For OM observation, the standard metallographic procedures were applied, including grinding, polishing and etching. The samples for OM were etched in a picral solution to reveal grain boundaries. The grain sizes were measured using the linear intercept method. The specimens for TEM observation were grinded to 80 μm, and then polished by 5 vol.% nitric acid ethanol solution using the twin-jet method. The