Materials Science and Engineering B 219 (2017) 28–36

Contents lists available at ScienceDirect

Materials Science and Engineering B

journal homepage: www.elsevier.com/locate/mseb

Influence of 45S5 Bioglass addition on microstructure and properties of ultrafine grained (Mg-4Y-5.5Dy-0.5Zr) alloy

K. Kowalski [a,*], M.U. Jurczyk [b], P.K. Wirstlein [c], J. Jakubowicz [a], M. Jurczyk [a]

[a] Institute of Materials Science and Engineering, Poznan University of Technology, Jana Pawla II 24, 61-138 Poznan, Poland
[b] Division of Mother's and Child's Health, Poznan University of Medical Sciences, Polna 33, 60-535 Poznan, Poland
[c] Department of Gynecology and Obstetrics, Division of Reproduction, Poznan University of Medical Sciences, Polna 33, 60-535 Poznan, Poland

ARTICLE INFO

Article history:
Received 5 December 2016
Received in revised form 14 February 2017
Accepted 27 February 2017

Keywords:
Magnesium
45S5 Bioglass
Mechanical alloying
Ultrafine grain

ABSTRACT

Bulk samples of an ultrafine grained (Mg-4Y-5.5Dy-0.5Zr)-x wt% 45S5 Bioglass (x = 0, 5) and (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass-1 wt% Ag composites have been synthesized by consolidating mechanically alloyed powders. The influence of the chemical composition on the microstructure, mechanical properties and corrosion behavior of bulk composites were studied. The sintering of (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass powders led to the formation of a bulk composite with grain size of approx. 95 nm. The corrosion behavior of Mg-based composites before and after hydrofluoric acid treatment was also investigated. The ultrafine grained (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass composite was more corrosion resistant than the bulk Mg-4Y-5.5Dy-0.5Zr alloy after HF treatment. The in vitro biocompatibility of synthesized composites was evaluated and compared with microcrystalline magnesium. Magnesium, (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass and (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass-1 wt% Ag composites modified with MgF_2 have a higher degree of biocompatibility in comparison with the unmodified reference material.

© 2017 Elsevier B.V. All rights reserved.

1. Introduction

Magnesium and its alloys have attracted a lot of attention as potential bone implant materials [1,2]. They are characterized by non-toxicity and biodegradability in physiological environment [2,3]. But, the lower corrosion resistance in chloride containing solutions is the main problem [4–6].

In the presence of a multiphase structure, magnesium alloys undergo localized corrosion due to galvanic effects [7]. The rare earth (RE) improve the mechanical properties and accelerates the kinetics of the MgH_2 formation, which prevents further dissolution of magnesium alloys [8]. Due to the grain refining effect, the addition of Zr improves not only the mechanical properties but also the corrosion resistance of magnesium alloys [9].

Several investigations have been done to study the effect of rare earths when they are alloyed to pure magnesium and other Mg alloys [10]. For example, addition of yttrium as an alloying element in Mg has been tested owing to the fact that there exists a large difference in the atomic radii of Mg (145 pm) and Y (212 pm) which allows strengthening of Mg by both solid solution strengthening and precipitation-strengthening. It was reported that Y with max solubility of 4.7 at.% in Mg can effectively strengthen Mg by solid solution strengthening [11]. On the other hand, dysprosium has a high solid solubility in Mg [12]. Mg-10Dy alloy can be developed further for biomedical applications due to its mechanical and corrosion properties [13].

Silver (Ag) was shown to have an antibacterial effect and can also enhance the mechanical properties of magnesium alloy [14]. Cytotoxicity of Ag nanoparticles at a concentration of 8 mg/cm^2 to *E. coli* was reported [15]. Silver nanoparticles provides good antibacterial behavior [16]. Mg–Ag binary alloys with up to 6 wt % of Ag in Mg have been investigated in vitro and in vivo [17,18]. The Mg–2 wt% Ag shows adjustable improved mechanical, corrosive, cytocompatible and antibacterial properties.

An improvement of the corrosion rate of Mg-based alloys can be achieved also by surface treatment processes such as chemical conversion coating [19], layered double hydroxide coating [20], micro-arc or plasma electrolyte oxidation [21], layer-by-layer film [22] and polymeric coating [23]. The mechanical surfacing, such as machining, deep rolling or low plasticity burnishing, can be applied as well [24].

New perspectives appear with nanostructured or ultra-fine grained materials, which exhibit better physico-chemical, biological and mechanical properties in comparison to their microcrystalline counterparts [25]. Usually, these biomaterials were

* Corresponding author.
E-mail address: kamil.kowalski@put.poznan.pl (K. Kowalski).

http://dx.doi.org/10.1016/j.mseb.2017.02.010
0921-5107/© 2017 Elsevier B.V. All rights reserved.

produced by the application of non-equilibrium processing techniques, such as mechanical alloying (MA) or severe plastic deformation (SPD) [26,27]. Recently it has been reported, that the mechanical properties of the Mg-based composite can be improved by the addition of hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) nanoparticles [28]. Our latest studies have focused on the possibility of HA application in a composite form, in materials uniting Mg-4Y-5.5Dy-0.5Zr with hydroxyapatite [29].

The most common ceramics, used in medicine except HA is 45S5 Bioglass [30,31]. The bioactivity of bioglass is attributed to the formation on their surface of a hydroxycarbonated apatite (HCA) layer similar, to a large extent, to the mineral part of the bone [32]. 45S5 Bioglass has the potential to be used in many more bioactive applications than hydroxyapatite [33].

Recently, Witte et al. have studied the properties of a metal matrix composite (MMC) composed of magnesium alloy AZ91D as a matrix and hydroxyapatite particles as reinforcements [34]. The mechanical properties of the MMC-HA were dependent on the HA particle size and distribution.

In our previous studies, the mechanical alloying method and the powder metallurgy process for the fabrication of ultrafine grained (Mg-4Y-5.5Dy-0.5Zr)-x wt% HA composites with a unique microstructure have been developed [29]. For example, for the bulk (Mg-4Y-5.5Dy-0.5Zr)-5 wt% HA composite and (Mg-4Y-5.5Dy-0.5Zr)-5 wt% HA scaffold with 48% porosity, the average Young's modulus of 29.3 GPa and 7.1 GPa was measured, respectively. This project provided the first evidence for an enhancement of the properties due to the ultrafine grained structures in consolidated (Mg-4Y-5.5Dy-0.5Zr)-HA composites.

As the continuation of the previous work, the present study examines the microstructure, mechanical and corrosion properties as well as in vitro cytocompatibility of the bulk ultrafine grained Mg-4Y-5.5Dy-0.5Zr material through 45S5 Bioglass alloying. The corrosion behavior and in vitro cytocompatibility of synthesized composites before and after the hydrofluoric acid (HF) treatment was investigated, too. The properties of the bulk ultrafine grained (Mg-4Y-5.5Dy-0.5Zr)-x wt% 45S5 Bioglass (x = 0, 5) and (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass-1 wt% Ag composites have not been previously investigated.

2. Experimental

2.1. Chemicals and materials

The following commercial powders were used: magnesium (<45 μm, 99.8%, Alfa Aesar), zirconium (<350 μm, Ciech-Poland), silver (4–7 μm, 99.9%, Alfa Aesar), 45S5 Bioglass (45% SiO_2, 24.5% Na_2O, 24.5% CaO, 6% P_2O_5, 53 μm; all in wt% from Mo-Sci Health Care L.L.C., USA). Powders of yttrium and dysprosium (about 100 μm in size) were prepared from the bulk samples (99.9% purity; Alfa Aesar) via mechanical filing approach in argon atmosphere.

The experiments were carried out on the Mg, Mg-4Y-5.5Dy-0.5Zr alloy, (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass and (Mg-4Y-5.5Dy-0.5Zr)-5 wt% 45S5 Bioglass-1 wt% Ag composites before and after immersion for 2 h in 40% HF. For brevity, in this work, materials are denoted as follows:

- Mg-4Y-5.5Dy-0.5Zr ultrafine grained alloy is labeled as Mg9.5RE0.5Zr,
- Mg-4Y-5.5Dy-0.5Zr –5 wt% 45S5 Bioglass composite is labeled as Mg9.5RE0.5Zr 5BG,
- Mg-4Y-5.5Dy-0.5Zr –5 wt% 45S5 Bioglass composite after immersion for 2 h in 40% HF is labeled as Mg9.5RE0.5Zr 5BG HF,
- Mg-4Y-5.5Dy-0.5Zr –5 wt% 45S5 Bioglass–1 wt% Ag composite is labeled as Mg9.5RE0.5Zr 5BG1Ag,
- Mg-4Y-5.5Dy-0.5Zr–5 wt% 45S5 Bioglass–1 wt% Ag composite after immersion for 2 h in 40% HF is labeled as Mg9.5RE0.5Zr 5BG1Ag HF.

2.2. Sample preparation

Mechanical alloying was carried out using a 8000 SPEX mixer mill, employing a weight ratio of hard steel balls to powder weight ratio of 20:1 at ambient temperature for 40 h in a continuous mode. The elemental powders were weighed, blended and poured into a round bottom stainless vials in a glove box (Labmaster 130) filled with automatically controlled argon atmosphere ($O_2 \leq 2$ ppm and $H_2O \leq 1$ ppm) to obtain the nominal Mg9.5RE0.5Zr, Mg9.5RE0.5Zr 5BG and Mg9.5RE0.5Zr 5BG1Ag compositions.

The bulk samples were prepared using powder metallurgy. The powders were uniaxially pressed at a compacting pressure of 600 MPa. The typical dimensions of the pellets were d = 8 mm in diameter and h = 3 mm in height. Finally, the green compacts were sintered at 550 °C for 2 h under argon atmosphere (99.999% purity) to form the bulk samples.

Fluoride treatment is a commonly used technique to optimize the degradation kinetic and improve the biocompatibility of magnesium-based implant [35]. It has good bonding strength and can be performed on complex structures such as stents. Generally, Mg-based samples were suspended in 40% HF for some time (up to 24 h) to produce a fluoride coating. In this work, for surface HF treatment, the polished samples (1000 grit SiC paper) were immersed in 40% hydrofluoric acid for 2 h at a room temperature. A compact, tight and uniform coating mainly composed of MgF_2 was formed on the surface of each sample. Finally, the samples were washed with distilled water, dried and subsequently assessed visually for the uniformity of the coating layer.

2.3. Material characterization

The structure, microstructure, composition and morphology of the materials were studied by X-ray diffraction and scanning electron microscopy. SEM with the energy dispersive spectrometer was used to characterize the chemical composition of the prepared samples.

The density of the sintered samples was determined by Archimedes method. The porosity of the synthesized materials was calculated by the formula $P = (1 - \rho/\rho_{th}) \times 100\%$, where ρ and ρ_{th} are the density of the synthesized material and its corresponding theoretical density.

The microstructure of the bulk synthesized materials was studied by the Quesant Q-Scope 250™ atomic force microscope (AFM). The Vickers microhardness ($HV_{0.3}$) of the bulk samples was measured using a microhardness tester by applying a load of 300 g on the polished surfaces of the samples. Indentation Hardness (HV) and Young modulus (E_{IT}) of the non-etched Mg9.5RE0.5Zr and Mg9.5RE0.5Zr 5BG materials, was evaluated using a CSM Instruments nanoindenter with the Berkovich diamond tip [36]. Depth-sensing indentation technique were used for measurements of:

- indentation Hardness (HV) that is defined as the mean contact pressure, that is the indentation load divided by the projected area of contact,
- indentation Modulus (E_{IT}) testing calculated from the slope of the tangent for the calculation of indentation hardness following the method given by Oliver and Pharr [37], carried out on the samples by four-sided Vickers diamond indenter with an ISO 14577 standard for measurement parameters as follows F = 0.3 N/20 s and C = 5.0 s. The all bulk samples to nano

Download English Version:

https://daneshyari.com/en/article/5448803

Download Persian Version:

https://daneshyari.com/article/5448803

Daneshyari.com