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Full Length Article

Apatite formation and weight loss study in EDMed perforated AZ31 Mg-alloy

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Abstract

Porous structures are highly preferred for bone regeneration and high tissue in-growth. In present work, electrical discharge drilling (EDD), a thermal erosion process was used to produce through holes in Mg-alloys to fabricate perforated structure similar to open cell porous structure in extruded AZ31. Apatite formation and weight loss study was conducted for 7 days, 14 days and 21 days after immersion tests in SBF solution. The perforated structure in AZ31 with 26 through micro-holes provides 72% increase in surface area but with marginally 4% higher weight loss as compare to non-perforated structure. Comparing perforated and non-perforated samples of Mg-alloy, it was well observed that perforated structure forms high volume of apatite as compared to non-perforated structure. Scanning electron microscopic (SEM) study revealed that in perforated structure, drilled holes retain their circularity after 21 days of immersion test and distinct corrosion phenomenon occur at localized sites.

Keywords: Mg-alloys; AZ31; Perforated structure; Electrical discharge drilling; Apatite formation; Weight loss

1. Introduction

Biodegradable implants are now being given high attention due to good bio-resorption, non-inflammatory reactions and stimulated bone growth [1]. Magnesium (Mg) and its alloys are easier to process with conventional machining methods, having low density, high specific strength, modulus of elasticity comparable to that of bone and stimulate bone regeneration [2,3]. In consequence, it is a potential candidate as implant materials. Though the biodegradable implants are allowed to dissolve, the material still need to possess the desired strength at least until it has served its purpose. Hence, for orthopedic applications, in addition to corrosion rate, the in- service mechanical integrity of the implant is also a critical factor. But Mg and its alloys, corrode rapidly in saline media such as human body environment, thus affects the mechanical stability with fast reduction of bending strength [4,5].

The degradation or corrosion rate can be controlled through suitable alloying, modifying the metallurgy of the alloy and surface modification [6]. Element alloying plays the most important role on the manipulation of corrosion resistance of biomedical Mg alloys [7]. In recent times, a considerable kind of novel magnesium alloys such as Mg-Ca [8], Mg-Zn-Ca [9,10], Mg-Mn-Zn [11], Mg-Zn-Si-Sr [12], Mg-Zn-Mn-Ca [13] and Mg-RE [14], have been investigated for the purpose of biomedical applications. Significant amount of work has been carried out on aluminum (Al) containing alloys like AZ91, AZ31 etc. and rare earth containing alloys [15] where the biocompatibility of these alloys has been questioned. Corrosion studies have been conducted on surface modification through Ca-P or HA coating [16], chemical conversion coating [17], polymeric coating [18] and micro-arc oxidation [19] etc. Corrosion resistance increases with these surface modifications [20] but several issues have also been reported such as adhesion between Ca-P coatings and their substrates, biocompatibility of the chemical conversion coatings, swelling of coating etc.

Porous structures are of high interest for tissue engineering for bone regeneration; as it allows high tissue in-growth, firm bone-implant fixation, permit the flow of body fluid etc [21]. Space holder technique is commonly used to fabricate open cell porous Mg-structure [22]. Pore size and porosity in porous Mg-alloy are key factors affecting the strength of the structure, tissue in-growth and degradation rate. Using micro-drilling,

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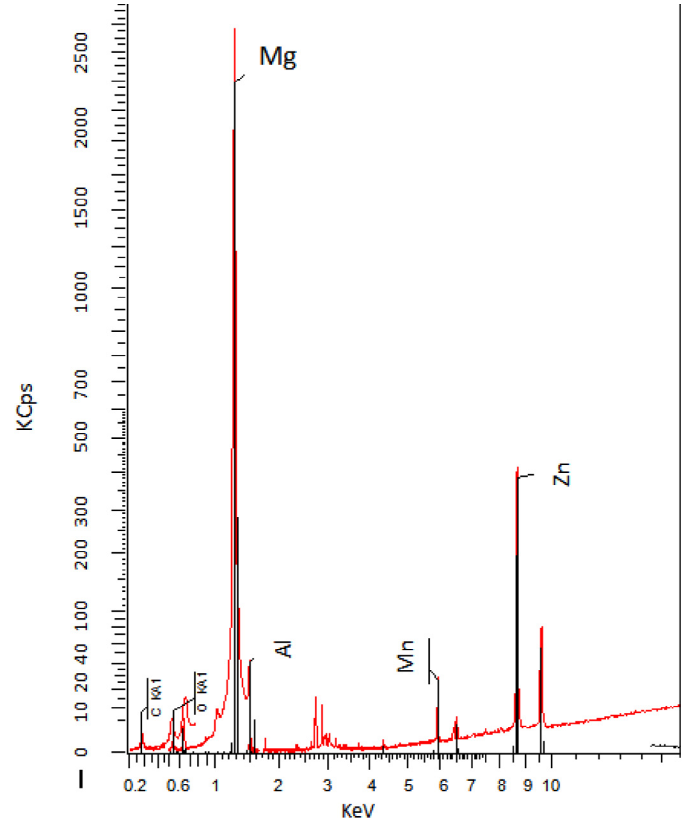

Fig. 1. XRF spectra of AZ31.

interconnected micro-holes can be produced in Mg-alloy to produce open cell structure similar to porous scaffold.

In present work, electrical discharge drilling (EDD), a thermal erosion process was used to produce high aspect through holes in Mg-alloy with 500 µm diameter. To vary the surface area, 13 and 26 number of holes were drilled separately in extruded AZ31 Mg-alloy. Thermal erosion process helps to generate fine surface with surface roughness between 1 to 2 µm. This micro roughness would help to attract cell tissues on biomaterials. The immersion test were carried out in Simulated body fluid (SBF) solution at 37^0C for 7 days, 14 days and 21 days to study the apatite formation and corrosion in saline fluid. The corrosion study was conducted in terms of weight loss of the samples after regular interval of time. Scanning electron microscopic (SEM) analysis was also carried out for degraded samples to study the corrosion morphology. Elemental dispersion spectroscopy (EDS) analysis was conducted to determine the elements dissolved in the apatite formed during immersion tests.

2. Materials and methods

2.1. Material preparation

In the present work commercially available AZ31 Mg-alloy was used as a work material in the form of extruded rod of diameter 40 mm. The chemical compositions of supplied Mg-alloy (wt. %) was determined using XRF (Fig. 1) as Zn: 2.59%, Al: 0.98%, Mg: 95.79%.

To examine the microstructure of Mg alloys rectangular samples of 10 × 10 mm was polished using 400, 800, 1000 and 2000 grit paper, ultrasonically cleaned in acetone and distilled water, then dried in open air. The samples were etched using an etching agent solution of 5 ml HF, 20 ml Nitric Acid, 20 ml HCl and 60 ml water. The microstructure was observed using an inverted metallurgical microscope as shown in Fig. 2a-b.

2.2. Electric discharge drilling (EDD)

EDD is a non-contact thermal erosion process for producing micro holes in metallic materials [23]. Using EDD interconnected micro holes can be easily fabricated in bio-metallic implants to produce perforated structure similar to porous structures for better tissue growth and firm bone to implant fixation.

In present work 3- axis EDD machine (shown in Fig. 3) with copper tubular electrode of diameter 500 µm was used to obtained through micro holes in polished Mg alloy. Perforated and non-perforated AZ31 samples were obtained in the size of 20 mm x 20 mm x 4 mm as shown by computer aided drawing in Fig. 4a-b. Table 1 lists the size, weight and surface area of non-perforated and perforated Mg alloy samples.

In perforated samples, the surface area increases as compare to non-perforated sample. Surface area is calculated as; for non perforated samples (X): $2 \times (20 \times 20) + 4(20 \times 4) = 1120\ mm^2$; for perforated samples (Y): $(X\text{-}2\pi r^2 + 2\pi rh)$, where; r: radius of drilled hole (250 µm), h: depth of hole (20 mm).

3. Results and discussion

In present work, perforated and non-perforated rectangular samples were prepared that were polished at the outer surface.

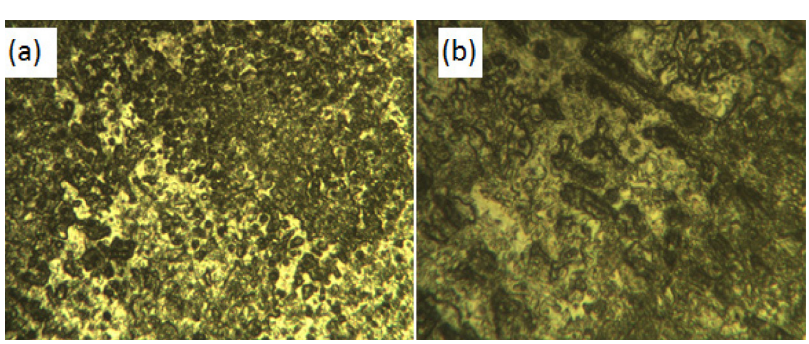

Fig. 2. Microstructure of AZ31 (a) at 10x (b) at 20x.