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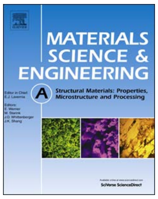

Reinforced magnesium composites by metallic particles for biomedical applications

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ABSTRACT

Pure magnesium (Mg) implants have unsatisfactory mechanical properties, particularly in loadbearing applications. Particulate-reinforced Mg composites are known as promising materials to provide higher strength implants compared to unreinforced metals. In the current work biocompatible niobium (Nb) and tantalum (Ta) particles are selected as reinforcement, and Mg-Nb and Mg-Ta composites fabricated via a powder metallurgy process associated with the ball milling technique. The effect of Nb and Ta contents on the microstructure and mechanical properties of Mg matrix was investigated. There was a uniform distribution of reinforcements in the Mg matrix with reasonable integrity and no intermetallic formation. The compressive mechanical properties of composites vary with reinforcement contents. The optimal parameters to fabricate biocompatible Mg composites and the optimal composition with appropriate strength, hardness and ductility are recommended.

1. Introduction

Magnesium (Mg) and Mg alloys have relatively low density and a high specific ratio of strength and weight. These specifications form the basis for commercial. Mg and its alloys have found successful use in a wide variety of applications such as nuclear, automotive, aircraft, shipbuilding, pipeline and electronic industries [1–3]. Recently, Mg and its alloys are used increasingly as a promising biodegradable material for medical applications according to their analogous mechanical properties to bone tissue, functional roles in the human body, good biocompatibility and a higher strength to weight ratio rather than other metallic and polymeric biomaterials [4,5].

However, some disadvantages such as low strength, high degree of shrinkage during solidification of molten Mg, high chemical reactivity especially at elevated temperatures have limited the use of Mg [6]. Also, pure Mg has poor mechanical performance particularly in orthopedic applications. Mg may corrode too quickly in physiological and high chloride environments which leads to loss of mechanical integrity before sufficient healing of tissues [5,7].

Alloying, is one of the most common strategies to improve the mechanical properties as well as corrosion performance of metallic Mg [8]. So far, various alloying elements have been used and extensive investigations have been carried out on the composition design. But, most of the incumbent Mg alloys exhibit a certain degree of cytotoxicity and low corrosion resistance. Moreover, they possess low mechanical properties which are further reduced during degradation. Thus, they cannot mechanically endure and tolerate the activity of the patient for a significant time after implantation [4,7,9–11].

The addition of reinforcements to Mg matrix is an alternative technique, and this introduces a composite structure, resulting in improved mechanical properties, corrosion performance and wear resistance. High flexibility in component design and reinforcement material in composites also can rectify the biocompatibility of Mg [1,12,13]. Various types of reinforcements have been used in the Mg matrix over the past decades. Continuous fibers were early materials used as reinforcement in Mg matrix; however, they were then substituted by discontinuous fibers and whiskers due to some restrictions such as cost of continuous fibers and limited fabricability and availability. Particles are receiving increasing attention as an alternative type of reinforcement and are currently used commercially.

Mg composites reinforced with ceramic particulates such as carbon nano tubes, Al_2O_3, SiC and graphite or metallic particles such as titanium, niobium, nickel and copper have been investigated [14–20]. The choice of reinforcement is dictated by several factors such as application, manufacturing method and cost of fabrication [14,21,22]. In medical applications, the biocompatibility and biodegradability of reinforcements needs to be considered. A few biocompatible reinforcements such as calcium, calcium polyphosphate, hydroxyapatite, fluor-

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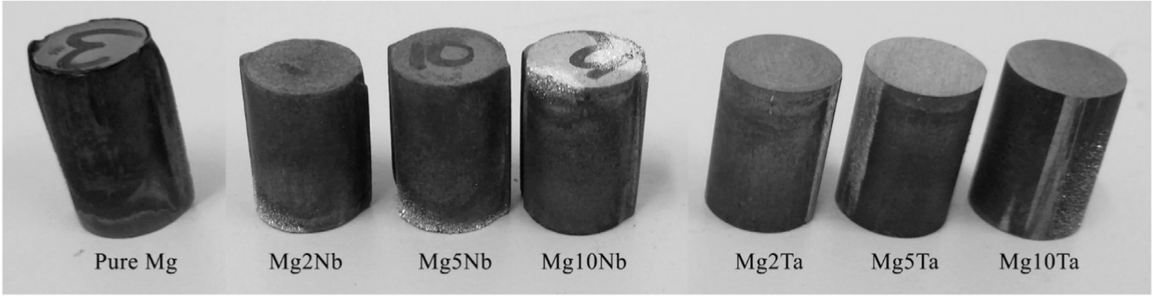

Fig. 1. Bulk sintered pure Mg, Mg-Nb and Mg-Ta composites fabricated through powder metallurgy process.

apatite, titanium, niobium, have been used so far, while the properties and interaction of some with a Mg matrix have not been comprehensively investigated [13,14,23–26].

In addition, the reinforcements, matrix materials and properties of composites are very sensitive to fabrication method. There are two major methods to produce particle-reinforced Mg composites: powder metallurgy (PM) and molten methods. In the molten method, some parameters such as high activity of molten Mg, lack of wettability of some particles by molten Mg and reactivity of some reinforcements with molten Mg have to be taken into consideration. However, the powder metallurgy method, implemented at low temperatures, is appropriate for highly reactive and low wettable reinforcements and Mg matrix. Moreover, the higher flexibility in reinforcement material, feasibility to utilize high volume fractions of reinforcements of up to 50% and higher available homogeneity have developed PM as a promising technique in Mg composite fabrication [6,21,27].

The Mg matrix can be reinforced with biocompatible niobium (Nb) and tantalum (Ta) particles through PM process. A few reports, to author's knowledge, about Mg-Nb or Mg-Ta composites are available from the literature. High strength Mg-Nb nanolayer composites have been fabricated through sputter deposition by Ham and Zhang [28]. Shanthi and co-researchers fabricated Mg-Nb composites through the molten method [14]. They observed a uniform distribution of Nb particulates in the Mg matrix with no formation of intermetallics. Improved mechanical properties of Mg-Nb composites with addition of Nb up to 15% (wt% hereafter) have been reported. There, Mg5Nb was the optimal composition according to the strength and ductility.

In this study, Mg composites are reinforced with biocompatible niobium (Nb) and tantalum (Ta) particles through the PM process. The effect of biocompatible Nb and Ta particulates on the microstructure of Mg matrix are investigated. Moreover, the mechanical properties of composite materials are examined via microhardness and compression test. The optimal parameters to fabricate Mg composites via powder metallurgy process are recommended.

2. Materials and method

Commercially available Mg powders (purity: ≥99.9%, 60≤ size ≤220 µm, Parameet Co. ltd., South Korea), Nb powders (purity ≥99.9%, 5≤ size ≤120 µm, Atlantic Equipment Engineers, USA) and Ta powders (purity ≥99.9%, 2≤ size ≤15 µm, Atlantic Equipment Engineers, USA) were used as starting materials. Mixture of Mg-Nb or Mg-Ta powders at a nominated ratio of Mg and Nb or Ta powders were prepared by high energy ball milling. The ball milling was performed with a planetary ball mill PM 400-Retsch under argon protective atmosphere for 9 h. The ball-to-powder ratio was 20:1 and ball milling was carried out at rotation speed of 200 rpm. The milled pure Mg powder, as control material, and homogeneous mixture of Mg-Nb or Mg-Ta powders were uniaxially pressed at a pressure of 760 MPa into cylindrical compact. The green compacts were then sintered at 610 °C for 3 h. The density of pure Mg and composites was measured using the Archimedes' Principle. Three randomly selected samples were weighted in air and distilled water, using an electronic balance, with an accuracy of $\pm 0.0001g$.

The sintered samples were then grounded, using 1200 grit SiC paper, and micro-hardness test was carried out using Highwood HWDV-7S microhardness tester accordance with ASTM E384-99 standard [29]. The x-ray powder diffraction (XRD) was performed on a PANalytical X-pert Powder diffractometer (Netherlands) with CuKα radiation. An Olympus optical microscope and Zeiss Supra 55VP scanning electron microscope (SEM) were used to characterize the powders and microstructure of composites. To determine the porosity and pore size distribution, sintered samples were cut transversely with a diamond wafering blade. The cut surfaces were then polished to 4000 grit SiC paper finish. The SEM images and commercially available software ImageJ and Lineal Analysis (LA) method were used for quantitative measurement of porosity and pore size distribution [30]. Three samples of each composition were cut into five cross sections (i.e. fifteen sections were considered) to obtain a reliable statistic of porosity and pore size distribution. Finally, Instron universal tester (Instron 5567, 30 kN, USA) was used for compression test of cylindrical samples with a size of $\Phi 10\times 15\ \text{mm}^2$ with an initial strain rate of $10^{-3}\ \text{s}^{-1}$. A total of 9 samples were tested for each composition.

3. Results and discussion

As can be seen in Fig. 1, Mg-Nb and Mg-Ta composites were fabricated successfully through powder metallurgy process. Pure Mg was also fabricated as a reference material. The samples were then cross sectioned and the microstructures were investigated using SEM (Fig. 2). Some micro-pores are observed in the microstructures. The presence of porosity is due to the trapped air between Mg particles which is related to the fabrication method i.e. it is the normal characteristic of powder metallurgy process. It also could be related to the volume shrinkage of Mg powders [31]. However, the porosity in current samples is agree with and even better than other PM-processed Mg composites [32]. Table 1 represents the density and porosity of current pure Mg and Mg composites. Rule-of-mixture (ROM) approach (Eq. (1)), based on the values of density (ρ_{Mg}=1. 739, ρ_{Nb}=8. 57, ρ_{Ta}= 16. 69 g/cm^3) and volume fraction of metal matrix and reinforcements, was used to calculate the theoretical density of composite materials [33–35].

$$\rho_{composite} = \rho_{matrix} \times V_{matrix} + \rho_{reinforcement} \times V_{reinforcement} \tag{1}$$

Where, ρ is density (g/cm^3) and V is volume fraction (vol%). The low differences between theoretical and experimental densities approve the feasibility of fabrication of almost dense pure Mg and Mg composites through PM and milling processes. The low porosities, measured by lineal analysis (LA), as well as small pores in the cross sections also demonstrate dense structure in fabricated pure Mg and Mg composites.

In current research, high pressure and sintering temperature used to consolidate Mg powders to green compact and attach particles together, respectively. Thus, low enclosed air was entrapped between powders, resulted in low porosity in the microstructures. The transfig-