



Processing of magnesium foams by weakly corrosive and highly flexible space holder materials



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ABSTRACT

High-quality magnesium foams were fabricated by an infiltration technology using tailor-made salt–flour mixture space holders. The pore structures and mechanical properties of space holder particles as well as the resultant foam production with spherical pores were characterized in the present study. The particles after high-temperature sintering dissolved rapidly in water due to their porous structures, guaranteeing the weak corrosion and high-purity of magnesium foams. The spherical pores foams exhibited usual stress–strain behaviors and nearly isotropic properties. The yield strengths of the foams increased with the decrease of sample porosity, and the relative mechanical properties of foams were mostly dependent on their relative densities, which obeyed a power law relation. Moreover, porous magnesium materials with tunable pore structures could be fabricated owing to the flexible forming features of salt–flour mixture, showing great application prospects in bone implant material field.

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1. Introduction

Magnesium (Mg) foams have drawn much attention as promising biodegradable bone implant materials due to their outstanding biocompatibility, favorable mechanical properties and similar porous structures close to that of natural bones [1]. In order to produce Mg foams suitable for bone implants, many techniques have been developed including powder metallurgy [2–7], molten metal infiltration [8–12], metal foaming [13,14], metal/gas eutectic unidirectional solidification method [15], and so on. These technologies do have yielded Mg foams with a variety of pore structures and desired properties, but there still exist limitations and shortcomings. For example, in the first two approaches, the residues of resolvable space holders or porous preform materials are usually inevitable in the cell walls that give rise to the pollution and deteriorated physical/chemical properties of Mg foams. Wen et al. found that a little amount of carbon remained on the cell wall surface of Mg foams when preparing them using carbamide particles as the space holder [2]. However, the carbon residues would seriously impair the biocompatibility of the foams [1]. Moreover, even though with the same porosity the Mg foams showed noticeably different compressive flow stresses in the plateau region due to

the uneven distribution of the different sized pores in the samples [3]. In order to overcome these shortcomings, Hao et al. used round-shaped carbamide particles to substitute those polygonal ones and introduced a preliminary low-temperature consolidation and dissolution process prior to high-temperature sintering [4]. This process effectively eliminated the residues of space holder particles in the cell walls and improved the quality of Mg foams. However, the foams still presented brittle characteristics.

High-quality metal foams have often been obtained through infiltration technology. In the molten metal infiltration process for fabricating aluminum foams, NaCl (crude salt) particles are the most widely used preform material [16]. However, this material would be unsuitable for the preparation of Mg foams because of its strong corrosion effect as well as the high chemical activity of Mg matrix. Hence, it is necessary to find other preform materials of weak corrosiveness or shorten the dissolution time of preform to reduce the corrosion of Mg matrix. Xu et al. [10] adopted MgSO₄ to substitute NaCl in fabricating Mg foams via infiltration process and found that the corrosion of Mg matrix was reduced indeed, but the dissolution time was not obviously shortened, which was not suitable for the fabrication of large-scale Mg foam samples. Staiger et al. utilized 3D net shaped polymeric materials as the cell wall pattern filled with NaCl paste to prepare topologically-ordered Mg foams through burning-out the polymer, sintering the NaCl particles and infiltrating molten Mg [11,12]. The resultant Mg foams had very high purity and underwent quite weak corrosion due to quick dissolution of NaCl preform. These researches

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demonstrate that the employment of easily soluble space holders is an effective way to fabricate high-purity Mg or Mg alloy foams. In consideration of biomedical applications, it is of great value to obtain open-cell Mg foam with superior purity to ensure its biocompatibility and predictable mechanical performance to fulfill required load bearing functions during the implantation period. Although the Mg foams produced by the aforementioned metal foaming and metal/gas eutectic unidirectional solidification technologies have no harmful residues, they are not suitable for bone implants due to the inconsistent morphologies with closed cell structures [1,13,14].

Recently, a new method has been developed in fabricating aluminum foams using a soluble mixture of flour and salt powder as the space holder [17]. The preform can easily and quickly dissolve in water after the infiltration and thus should be also appropriate for fabricating Mg foams. The other advantage is the flexible shapes of space holder due to the easy forming characteristics of flour, being very beneficial to providing the metal foams with different pore structures and mechanical properties. Therefore, this technology was utilized in the present study with the objective of processing Mg foams with high-purity, spherical pores and favorable properties. The results are expected to provide useful information for the further studies on the development of Mg foams and their applications in the bone implant field.

2. Experimental details

Spherical salt–flour mixture particles were produced by granulation method [18], and the process is outlined schematically in Fig. 1. Specifically, crude salt was pulverized and then classified through a 200-mesh sieve. The resultant salt powders (powder size $\leq 74 \mu\text{m}$) and comparable sized flour with different contents (5, 10, 15 and 20 percent in weight ratio) were evenly mixed together. Finally, the spherical salt–flour mixture particles were acquired on a pan-type pelletizer utilizing spray water as the binder. After that, the spherical particles with the average diameter of 1.6 mm were obtained by sieving and then heated to 680°C for 2 h in air atmosphere to make the water volatilize and flour pyrolyze. Thus, the additives could be removed, which left behind the salt particles with high porosity (refer to Fig. 2a and b). The preform was subsequently prepared by stacking a number of salt particles under an appropriate axial compression in a cylindrical mould.

Mg foam was prepared by infiltrating molten Mg into salt preform followed by the dissolution of salt in ingot. Specifically, Mg ingot (99.7% purity) was placed on top of the mould and vacuum was pulled to 4×10^{-2} mbar residual pressure, and then the mould was heated to 680°C for 1 h before molten Mg was infiltrated into the preform using argon pressurized to 0.1 MPa. After solidification, the ingot was taken out and machined to the desired dimension prior to the removal of salt in 0.001 M NaOH water solution. The morphology of the resultant production is presented in Fig. 2c. The porosity of the sample was determined by the equation.

$$P = (1 - \rho_0/\rho_s) \quad (1)$$

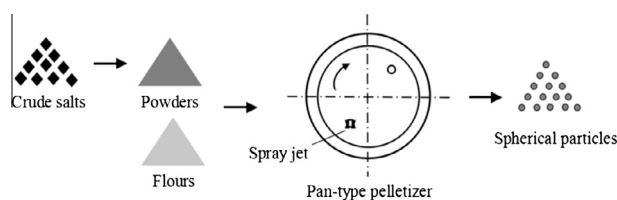


Fig. 1. Schematic illustration of the process for fabricating spherical salt–flour mixture particles.

where ρ_0 is the apparent density of the sample determined by its dimension and weight, and ρ_s is the density of Mg. The open porosity P_0 was estimated by the Archimedes method.

$$P_0 = (V_s - V_d)/V_s \quad (2)$$

where V_s and V_d are the samples' volume and drained water volume respectively.

The dissolution rate of spherical salt particles was characterized via an immersion test: 1 g specimen was immersed in 30 mL water and the crude salt was exerted for comparison. The morphologies of salt particles and foams were characterized on a field-emission scanning electron microscope (FE-SEM, FEI Sirion 200), and the surface profile of Mg foam was analyzed by EDS microanalysis system attached to FE-SEM. The compressive tests for salt preform and resultant foams were performed using an Instron 3369 materials testing system with a crosshead speed of 2 mm/min. The samples for the compressive tests were 20 mm in diameter and 20 mm in height. The elastic modulus of Mg foams were calculated on the basis of curves fitted to the linear elastic regions of the stress–strain curves and yield strength values were determined using the 0.2%-offset method.

3. Results and discussion

3.1. The structure and properties of spherical salt particles

Typical structure of salt–flour mixture particles fabricated by granulation method is shown in Fig. 2a. The particles are nearly spherical in shape and vary in size, and the diameter of the particle is centered largely in the range of 1.4–1.6 mm. The particles with desired size could be acquired by the optimization of granulation parameters. It was pointed out that the binder content and rotational speed as well as granulation time could affect the size distribution of granules [18]. In order to enhance the efficiency of granulation, the short granulation time (5 min), high rotational speed (240 rpm) and the appropriate spraying water (about 0.1 mm in diameter) were determined to fabricate spherical salt–flour mixture particles in the study. As can be seen from Fig. 2b, the single salt particle after high-temperature sintering appears obviously vesicular structure, except for the numerous cavities in spheroid mainly given by the volatilization of water and the burning-out of flour, the other reason could be attributed to the granulation process itself that combined fine powders into larger agglomerates without forming pressure, this would inevitably lead to the loose packing of salt–flour mixture powders (refer to the high magnification image inserted in Fig. 2b).

The dissolution rates of crude salt and spherical salt particles in water were characterized by immersion experiments. The time required for crude salt to dissolve was more than 15 min, while the complete dissolution of spherical salt particles with different flour adding (5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%) were about 45, 25, 20 and 20 s respectively. The dissolution rate of salt particles enhances with the increase of flour adding due to their high porosity, which was formed by the loose aggregation of fine salt–flour mixture powders, the volatilization of water as well as the flour pyrolysis. It was validated from Fig. 3a–d that many pores formed in the salt particles after high-temperature sintering. As observed from the image inserted in Fig. 3a that many sintering necks emerged on the border of interparticle contacts. Moreover, the densification was obviously discovered through visualized volume shrinkage after sintering. It was stated that boundary diffusion would dominate the sintering of NaCl powders with the size below $150 \mu\text{m}$ and lead to densification [19]. Therefore, the formation of sintering necks should be attributed to the boundary diffusion among fine salt powders. The prediction has been confirmed by

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