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Processing and characterisation of carbon nanotube-reinforced magnesium alloy composite foams by rapid microwave sintering

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ABSTRACT

The present study proposes an efficient processing scheme for fabricating carbon nanotubes (CNTs)-reinforced magnesium (Mg) alloy AZ61 composite foams with enhanced compressive and energy absorption properties. The scheme combines powder metallurgy, rapid microwave (MW) sintering, and pore wall reinforcement to overcome the low strength, non-uniform pore structure, prolonged sintering process, and high production cost associated with conventional unreinforced Mg-based foams. In the proposed scheme, a dual-stage mixing method is used to homogeneously disperse and incorporate CNTs into the matrix for strength enhancement, and susceptor role, and carbamide granules are used to control the pore size and porosity fractions. In addition, MW sintering is used to rapidly consolidate the samples in 20 min through the synergy between an external and an internal susceptor (i.e. CNTs), which facilitates uniform and volumetric heating of the entire samples. Thus, sample oxidation and the formation of deleterious secondary phases are minimised, while up to 69% energy is saved. Experimental results show that the dispersion and incorporation of CNTs into the matrix, via the present processing scheme, clearly enhance the compressive and energy absorption properties of the composite foams, as compared with the unreinforced foams. The proposed processing scheme is a rapid and energy-saving efficient technique, which can be used to fabricate high quality Mg alloy composite foams with improved compression and energy absorption properties.

1. Introduction

Metallic foams have high specific properties and are potentially useful for energy absorption, impact resistance, damping and lightweight structural applications. The lightweight characteristic of magnesium (Mg)-based foams possesses huge potential to increase fuel efficiency and reduce emissions for automotive and aerospace applications. Nevertheless, high production costs and processing difficulties are some of the problems encountered in the fabrication of Mg-based foams [1]. Furthermore, the low strength of Mg-based foams has majorly limited them to biomedical applications. As a result, Mg-based foams have not received the same research attention unlike aluminium (Al)-based foams which have been widely studied.

The melt-foaming route is commonly utilised for producing Mg-based foams. Although this processing route can produce high porosity foams, poor control over pore size and morphology are its prominent disadvantages [2]. Moreover, the variation in pore sizes and irregular cell shape can result in anisotropic mechanical properties [3]. Furthermore, prominent foaming/gas release agents, such as calcium hydride and titanium hydride, are expensive [4], while alternative agents, such as calcium carbonate ($CaCO_3$), release poisonous gases during decomposition at the foaming temperature [5]. Therefore, there is a need to find alternative solutions that are simple, cost effective and capable of producing high quality Mg foams.

The powder metallurgy (PM) processing technique is a relatively simple fabrication process that offers near-net shape production and can accommodate the incorporation of relatively high reinforcement fractions for fabricating metal matrix composites (MMCs) [6]. This technique has been successfully utilised to control the pore shape and size of a steel composite foam by using steel hollow spheres as spacing holding agents [7]. Similarly, some studies have shown that steel foams fabricated with spherical space holders possess higher compressive properties than foams having irregular pores [8]. Therefore, the PM technique can overcome some of the challenges encountered with using the melt processing technique.

Nonetheless, the consolidation of PM products is typically done through conventional sintering, which generally requires a prolonged sintering time of several hours. This increases the cost of production

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and the propensity for the formation of undesirable secondary phases. For instance, Mg foams were sintered for up to 48 h, which resulted in excessive grain growth, reduced porosity, and deleterious reactions between the base material and the silicon crucibles [9]. Other similar studies on Mg foams that required less sintering time in the range of 2 h [10] and 6 h [2] have reported a partial oxidation of the matrix. With the aim of harnessing the advantages of the PM processing technique for the fabrication of Mg foams, alternative rapid sintering methods, such as microwave (MW) sintering, have been developed. Successful MW sintering and improvement in the mechanical properties of AZ61 Mg composites [11] and biomedical properties of titanium composites [12] have been reported. Compared with conventional sintering, the advantages of MW sintering include rapid heating rates, volumetric heating, reduced sintering time and energy consumption [11,12].

Carbon nanotubes (CNTs) have low density, high strength, high elastic modulus, and high aspect ratio, which make them ideal reinforcement materials for both polymer [13–15] and metal matrices [11,16,17]. An improvement of 25–40% in the yield strengths of Mg-based composites reinforced with 1 vol% CNTs has been reported [16]. Similarly, Duarte et al. [17] report an increment of 100% in the Vickers microhardness of Al-foams reinforced with CNTs. Notwithstanding the property improvement reported in the above studies, achieving a homogenous dispersion of CNTs in the matrix and maintaining their structural integrity are some of the challenges encountered in the processing of CNT-reinforced MMCs [18]. Therefore, some fabrication techniques including ultrasonication processing [19] and mechanical milling [18] have been reported. Mechanical milling [20,21] has emerged as a good dispersion method, by which the structural integrity of the CNTs can be retained when the processing parameters are optimised [22]. In addition to the reinforcement effect of CNTs in the matrix, they could also serve as MW susceptors for enhancing volumetric heating, accelerating the heating rate, and reducing the overall sintering time [23].

Many previous studies on Mg-based foams have targeted orthopaedic implant applications, where relatively low strengths comparable to those of bone tissues are required [24,25]. Mg-based foams, with higher compressive strength and energy absorption capability, are required for high performance automobile or aerospace applications. A promising approach to address the low strength of Mg-based foams is to reinforce the Mg matrix with high strength particles, such as CNTs. As a result, the application areas of Mg-based foams could be broadened to different industries, such as automobile and aircraft. However, the synthesis of CNT-reinforced Mg-based composite foams by means of MW sintering has not been reported.

Therefore, this study proposes an efficient processing scheme, comprising powder metallurgy, rapid microwave sintering, and matrix reinforcement for the fabrication of Mg alloy AZ61 composite foams to improve their compressive and energy absorption properties. In the proposed scheme, spherical carbamide granules are used to simultaneously control the pore shape, size and porosity of the specimens. CNTs are selected to perform a dual function, serving as matrix reinforcement material and as an internal susceptor, for accomplishing volumetric heating. The MW sintering process is enhanced by the synergy between the internal and external heating, which results in rapid sintering, minimises the formation of unwanted secondary phases, and reduces the overall sintering time for cost and energy savings. The effect of CNTs on the compression and energy absorption behaviour of the foams are also investigated. The results reported in this study can provide useful knowledge in the design of lightweight structural materials using MW technology.

2. Experimental procedures

2.1. Materials

The materials used in this study include water atomised Mg AZ61 alloy powder (150 – 300 μm, Tangshan Weihao Powder Co., Ltd, China), multiwalled carbon nanotubes (CNTs, purity > 95 wt%; internal diameter: 3 – 5 nm; outer diameter: 8–15 nm; length: ~50 μm; Chinese Academy of Sciences, Chengdu Organic Chemistry Co., Ltd., China), commercially available spherical carbamide granules (0.2 – 0.9 mm) and Zwitterionic surfactant, 3-(N,N-dimethyl stearyl ammonio) propanesulfonate, (TCI (Shanghai) Chemicals, Development Co., Ltd, China). Mechanical milling was used to reduce the particle size of AZ61 to ~ 50 μm to enhance its MW absorbability. Details of the milling process have been reported in a previous work [11].

2.2. Synthesis of CNT-reinforced AZ61 composite foams

The CNTs used in this work are expected to perform a dual role, i.e., as matrix reinforcement and MW absorption, which can contribute to accelerating the sintering process [11]. CNTs were first purified and functionalised by refluxing in a 3:1 mixture of 98% sulphuric acid and 68% nitric acid. The dispersion procedure was as follows: 1 g of the surfactant powder was dissolved in 150 mL of ethanol and ultrasonicated at 27 °C for 15 min. After the complete dissolution of the surfactant powder in ethanol, 3 g of CNTs were added to the mixture, which was further subjected to ultrasonication for 20 min for the complete dispersion of CNTs in the solution. Stoichiometrically calculated amounts of AZ61 powder were added to the ethanol-CNTs-surfactant mixture, to make 1%, 2%, 3% and 5% volume fractions (vol%) of CNTs. The resulting composite mixture was designated as AZ61/*x*% CNTs, where *x* is the volume fraction of CNTs in the mixture. The AZ61/CNTs slurry was sonicated at 60 °C to disperse the CNTs on the alloy powder and to evaporate the ethanol. The composite paste was collected and dried under vacuum at 80 °C for 8 h. After drying, the composite powder was put in a milling jar with equal weight proportion of two different sizes of hardened stainless-steel balls (Ø 8 mm and Ø 15 mm) and milled at 300 rpm for 60 min. The ball-to-powder charge ratio was 10:1 and 30 vol% of cyclohexane was added as a process control agent to minimise cold welding and to increase powder yield. To assess the structural integrity of the CNTs after the mechanical milling treatment, Raman spectra were collected under ambient conditions using a microspectrometer (Horiba Jobin Yvon, HR800), utilising a visible red excitation laser source of 632.8 nm and a power of 50 mW.

With the CNTs dispersed on the surface of the AZ61 alloy powder, carbamide granules were added to the composite powder and further mechanically mixed for 10 min. About 2 mL of ethanol was added to moisten the surface of the carbamide granules and to minimise particle segregation during mixing. The AZ61/CNTs/carbamide mixture was put in a rigid cylindrical die and uniaxially pressed at 350 MPa for 2 min to form green samples. The green samples were immersed in room temperature DI water for 3 h to eliminate the carbamide granules and subsequently generate the pore structure. Porous green samples were dried at 150 °C for 5 h prior to MW sintering.

2.3. Microwave sintering of foam samples and X-ray diffraction (XRD) analyses

The MW furnace used for sintering was a continuously adjustable 3 kW multimode high vacuum cavity operating at 2.45 GHz (HAMiLab-HV3; SYNOTHERM Corporation). MW power for the sintering operation was varied between 100 and 350 W to maintain a steady sintering temperature. A susceptor-enhanced MW sintering setup was used to rapidly sinter the samples at 500 °C for 20 min under a flowing high purity argon gas to minimise sample oxidation. The green samples were enclosed in a cylindrical susceptor kiln lined with a mixture of silicon carbide and graphite (SiC/graphite). X-ray diffraction studies were carried out to identify the phases present in the AZ61 powder and the sintered foam samples using a Rigaku SmartLab 9 kW XRD equipment. The samples were exposed to Cu Kα radiation (λ = 1.54056 Å) at a