



Preparation of nacre-like composites by reactive infiltration of a magnesium alloy into porous silicon carbide derived from ice template



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ABSTRACT

Lightweight and high-strength AZ91/SiC composites with lamellar structures were successfully prepared by using freeze casting (ice-templating) and reactive infiltration techniques. The infiltration dynamics was measured and activation energy calculated to be 7.74 kJ/mol. The compressive and flexural strengths of the composites with 30 vol% initial solid load reached 743 ± 20 MPa and 599 ± 44 MPa, about 1.8 and 1.5 times of those of the AZ91 alloy, respectively.

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1. Introduction

The distinct “brick-and-mortar” structure provides nacre with high strength and toughness [1]. Inspired by nacre's fantastic structure and fabulous property, novel techniques are being developed to fabricate nacre-like materials [2–4]. Recently, Deville et al. [2] have adopted freezing as a flexible path to build sophisticated porous and layer-hybrid materials. Following this technique, Launey et al. [3] prepared the composites by infiltrating an Al–12.6 wt% Si alloy into a 36 vol% porous Al_2O_3 scaffold with lamellar structures at 1173 K using gas-pressure infiltration, which exhibited fracture (crack-growth) toughness of $40 \text{ MPa m}^{1/2}$ and bending strength of 300 MPa.

Up to date, the preparation of the nacre-like metal–ceramic composites is limited to the Al matrix and most of the studies employed high-cost pressure infiltration technique. For the Mg matrix, to our knowledge, there is no report. As known, Mg has lower density and higher specific strength and modulus than Al, receiving wide attention in recent years for potential structural applications. However, Mg has strong chemical reactivity and high vapor pressure and thus is easy to ignite and evaporate at elevated temperatures, making the preparation of Mg-matrix composites difficult. Nevertheless, our previous study revealed that Mg has very good wettability with silica [5], favoring the spontaneous

infiltration into silica-coated ceramic scaffolds. Based on this knowledge, in this work we prepared for the first time the lamellar Mg/SiC composites using freeze casting and reactive infiltration techniques.

2. Materials and methods

The raw ceramic materials used in this work were commercially available SiC powders with an average particle size (d_{50}) of $5 \mu\text{m}$ and a purity of 98.5 wt% together with 16.5 wt% Al_2O_3 , 1.5 wt% MgO and 2 wt% Y_2O_3 . They were added as sintering aids. Besides, 0.6 wt% CMC-Na was used as dispersant and deionized water as solvent.

First, water-based SiC slurry involving sintering aids with a total solid load of 30 vol% was ball-milled and then de-aired. Subsequently, the slurry was poured into Teflon molds to shape into cylinders with 18 mm in diameter and 25 mm in height by unidirectional freezing at $-20 \text{ }^\circ\text{C}$. After demolding, the cold SiC bodies were freeze-dried at $-50 \text{ }^\circ\text{C}$ in a 10 Pa vacuum for 24 h to remove the ice inside [6]. The resultant SiC bodies were then fired at $1100 \text{ }^\circ\text{C}$ in air for 2 h to form an oxide layer (silica) at the SiC surface and further sintered at $1500 \text{ }^\circ\text{C}$ in Ar atmosphere for 2 h. During the firing process, the heating and cooling rates were $5 \text{ }^\circ\text{C}/\text{min}$.

In order to determine optimum infiltration parameters, in-situ observation of spontaneous infiltration for a commercial AZ91

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(Mg–9Al–1Zn) alloy into porous SiC scaffolds was carried out. The AZ91 alloy was pre-placed on the SiC scaffold and the couple was heated in a high-purity (99.999%) Ar gas at 5 °C/min in a tube furnace, which was initially evacuated to a vacuum less than 5 Pa. During the heating and isothermal dwelling (at the temperature between 610 and 690 °C with an interval of 20 °C) process, the shape of the AZ91 alloy was monitored by a video camera through quartz windows to record its morphological change. The spontaneous infiltration dynamics was characterized by time variations in the alloy height and contact diameter.

On the basis of the in-situ observation results, the infiltration of the AZ91 alloy into the SiC scaffolds was performed at 650 °C with a holding time of 5 min. Fully infiltrated samples were readily achieved. The microstructures of the composites were observed using an optical microscope (Axio Imager A2m, Carl Zeiss, Germany) and a scanning electron microscope (SEM, Evo18, Carl Zeiss, Germany), and phases were identified by X-ray diffraction (XRD, D/Max 2500PC Rigaku, Japan). The compressive and three-point flexural strengths of the composites were measured by a universal material testing machine (Instron 5689 Corp., USA).

3. Results and discussion

Fig. 1 shows the microstructures of the SiC scaffold prepared by freeze casting. The sample displayed a well-developed lamellar structure in both longitudinal and transverse sections. Because SiC has a strong covalent bond and the atoms have low diffusion coefficients, it is difficult to achieve densification even sintering at a temperature of 2000 °C [7]. However, in the present work, the SiC surface was preoxidized at 1100 °C for 2 h, forming a SiO₂ layer, which may further react with the sintering aids during subsequent high-temperature (1500 °C) firing and thus greatly promote sinterability and enhance the strength of the scaffold. Nevertheless, many cavities were still observed in the SiC lamellae; namely, the ceramic layers were still far from densification.

By way of the preoxidation treatment, the spontaneous infiltration was readily achieved and the entire process can be divided into three stages (Fig. 2(a)): (i) alloy melting stage, (ii) inoculation stage and (iii) rapid infiltration stage. Because of the coverage of an oxide film at the Mg alloy surface, no obvious infiltration was observed in stages (i) and (ii). The decrease in the drop height and increase in the contact diameter in the end of stage (i) were virtually due to the collapse of the alloy after melting. During the inoculation stage (stage ii), the height of the

alloy and the contact diameter did not change. However, with the progressive evaporation of Mg, the oxide film was finally disrupted at some locations, thus paving the way for the spontaneous infiltration. The duration of the inoculation stage depends on temperature (Fig. 2(b)). Clearly, a higher temperature favors a larger Mg vapor pressure and thus faster disruption of the oxide film. As the clean liquid Mg alloy contacted the pre-oxidized SiC surface, rapid infiltration occurred, which is characterized by the sharp decrease in the height of the alloy drop together with a certain extent of liquid spreading (i.e., increase in the contact diameter as shown in Fig. 2(a)). The infiltration rate (v) can be roughly evaluated from the time dependence of the drop height (dH/dt). Note that the infiltration rate reached 10.2 mm/min at 650 °C and 14.3 mm/min at 690 °C (Fig. 2(c)); thus the whole sample with a height of 25 mm could be fully infiltrated by the alloy in no more than 3 min providing that the amount of the alloy was sufficient. Furthermore, according to the Arrhenius equation:

$$\ln v = -E_a/(RT) + \ln A, \quad (1)$$

where E_a is the infiltration activation energy, A is a constant, R is the gas constant, and T is the absolute temperature, we calculated $E_a = 7.74$ kJ/mol. This small value suggests easy activation for the infiltration of the alloy into the porous SiC scaffolds.

Fig. 3 shows the macro- and micro-structures of the infiltrating sample, particularly at the liquid infiltration front area. The sample could be characterized by three regions: (i) a fully infiltrated region, (ii) a partially infiltrated region and (iii) a non-infiltrated region. In the fully infiltrated region, the alloy not only distributed in the open channels but also in the cavities of the ceramic layers; whereas, due to the limited amount of liquid in the infiltration front area, the alloy was found only in the small cavities of the ceramic layers but not in the large open channels (Fig. 3(b)-2). The elemental mapping graphs (Fig. 3(c)) indicate that Mg had a selective distribution in the front area, whose position largely coincided with that of Si in the SiC layer, suggesting that the liquid alloy preferentially penetrated into the small cavities in the ceramic layer and then into the large open channels between the ceramic layers under the capillary force. This phenomenon can be explained by the Laplace equation:

$$P = 2\sigma \cos \theta / r, \quad (2)$$

where P is the capillary force, σ is the surface tension of the melt, r is the capillary radius and θ is the contact angle. In the case of $\theta \ll 90^\circ$ for Mg on SiO₂ [5], the smaller the capillary radius, the larger the capillary force, and as a consequence, the small cavities

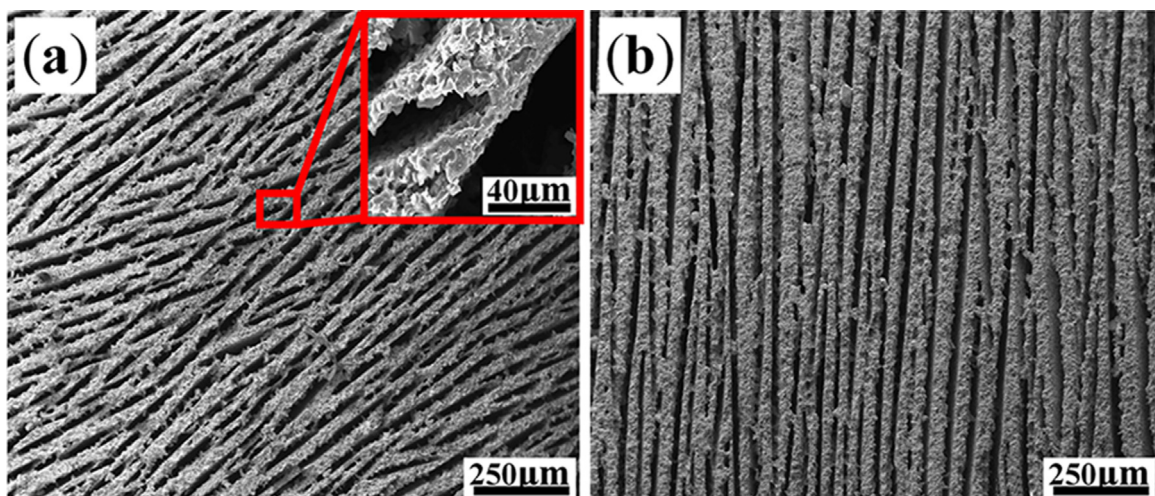


Fig. 1. SEM micrographs showing the lamellar structure of the SiC scaffold: (a) perpendicular to the freezing direction; (b) parallel to the freezing direction.

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