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Processing and mechanical properties of lamellar-structured Al–7Si–5Cu/TiC composites

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ABSTRACT

Inspired by nacre, we designed biomimetic metal–ceramic composites in which hard and ductile phases were alternately arranged. We first prepared lamellar-structured TiC scaffolds with 25, 30 and 35 vol% solid loadings using a unidirectional freeze casting technique and then measured wetting and infiltration dynamics for a commercial Al cast alloy, ZL107, with the main composition of Al–7Si–5Cu on the porous TiC scaffold using a modified sessile drop method at 850 °C. The wetting was affected by liquid spreading at horizontal surface and infiltration in depth, while the latter played a dominant role in the decrease in contact angle. On the basis of the wetting results, the lamellar ZL107/TiC composites were successfully fabricated by pressure infiltration of the molten ZL107 alloy into porous TiC scaffolds and their mechanical properties were evaluated. The compressive strength and elastic modulus increased while bending strength decreased with increasing TiC content, giving maximum values of 1166 ± 20 MPa, 205 ± 2 GPa and 363 ± 5 MPa, respectively. Formation of $(Al_m,Si_{1-m})_3Ti$, Al_3Ti and Al_4C_3 phases in the composites was presumed to deteriorate the toughness of the composites, leading to brittle fracture under loading.

1. Introduction

The distinct "brick-and-mortar" structure in nanoscale dimensions provides nacre with high strength and toughness [1]. Recently, inspired by the fantastic microstructure and fabulous properties exhibited in nacre, researchers have devoted to the development of the nacre-like materials using physical and chemical techniques such as layer-by-layer deposition [2], solution casting [3] and tape deposition [4]. However, these methods are restricted in preparation of the composites with only a few thickness (typically no more than 200 μm) that are not suitable for structural applications.

Recently, Deville et al. have developed a reliable method for the preparation of porous ceramic scaffolds by ice-templating (also termed freeze casting) [5]. The primary advantage of this method lies in the controllable structure and porosity of the scaffold as well as its great simplicity, economy and environmental friendliness. Using this technique together with gas-aided pressure infiltration, Launey et al. synthesized nacre-like Al–Si/Al_2O_3 composites, which exhibited high strength and toughness [6]. Likewise, Roy et al. and their colleagues prepared Al–12Si/Al_2O_3 composites by squeeze casting of the molten Al–12Si alloy into the lamellar-structured Al_2O_3 scaffolds and thoroughly investigated the elastic moduli [7–10], thermal expansion coefficient [11], internal load transfer mode [12], damage evolution and domain level anisotropy in the composites [13] by both experimental and theoretical analyses. Besides, Liu et al. prepared 2024Al/SiC composites by squeeze casting of the molten alloy into the lamellar-structured SiC scaffolds under a pressure of 80 MPa, followed by a T6 heat treatment. The highest flexural strength and fracture toughness (K_{IC}) were reported to be 931.3 MPa and 18.8 MPa·$m^{1/2}$, respectively, for the composites containing 10 vol% SiC [14]. In our prior work, we also fabricated similar-structured composites (Al–Si–Mg/Al_2O_3 [15] and Al–Si–Mg/SiC [16]) by way of pressureless infiltration.

It is noteworthy that all these previous studies focused on biomimetic synthesis of the materials and the resultant structures and properties, seldom concerning the wetting and infiltration behavior in the materials processing. As known to all, the wettability of ceramic by molten metal plays a crucial role in determining the ease of the infiltration and the interfacial bonding quality between the reinforcing phase and the matrix, thereby greatly affecting the final performance of the composites. On the other hand, from an academic point of view, the wetting of a highly porous scaffold by a molten metal involves liquid spreading and penetration, which may occur simultaneously, and thus how to evaluate the intrinsic wettability in such a complex case remains a challenge. Furthermore, to our knowledge, only very limited metal–ceramic systems have been selected as the candidate, mainly concentrating on the Al–Si eutectic alloy and alumina or silicon carbide. It is necessary to apply the biomimetic idea and advanced fabricating techniques to a broad range of material systems in order to obtain the composites with desirable strength and toughness.

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Based on the above considerations, in this work we selected ZL107 alloy (Al–7Si–5Cu) and TiC as a candidate system. The reason for the selection of TiC is due to its high strength and elastic modulus [17]. Also, as a transition metal carbide, it possesses relatively good wettability with metals such as Al and Mg [18]. The selection of the ZL107 alloy is because of its low density, low melting point, good fluidity, high strength and low cost. Moreover, this alloy is widely used in automotive industry [19–21]. In order to fabricate the nacre-like composites, we first prepared lamellar TiC scaffolds by using ice-templating. Then, we investigated the wetting dynamics between the molten ZL107 alloy and the lamellar TiC scaffold to determine the appropriate infiltration route. Finally, we fabricated the lamellar-structured ZL107/TiC composites by using gas pressure infiltration and examined the microstructure and mechanical properties of the resultant composites.

2. Experimental procedure

2.1. Preparation of lamellar TiC scaffolds

Commercial TiC powders with diameters of 2–3 μm and a purity of 98.5 wt% were used as raw materials to prepare the lamellar TiC scaffolds with initial solid loadings of 25, 30 and 35 vol%. In addition, 0.5 wt% sodium carboxymethyl cellulose (CMC-Na) based on total powder contents were added into water-based slurries as dispersant. The water-based TiC slurries were first ball-milled for 12 h and then deaired by stirring in a vacuum desiccator for 20 min. Subsequently, the slurry was poured into Teflon molds to shape into cylinders with 30 mm in diameter and 30 mm in height by unidirectional freezing through a Cu bar, whose top surface was controlled at −20 °C and bottom immersed in liquid N_2 [15]. After demolding, the cold TiC bodies were freeze-dried at −50 °C in a 10 Pa vacuum for 48 h to remove the ice inside. The resultant TiC bodies were then sintered at 1200 °C in a vacuum about 100 Pa and further at 1500 °C for 2 h in a flowing Ar atmosphere (99.999% purity). During the sintering process, the heating and cooling rates were 5 °C/min.

2.2. Wetting measurements

We first used a modified sessile drop method [22] to measure the wetting and infiltration behavior for the molten ZL107 alloy on the TiC scaffold (30 vol%) in a vacuum (1×10^{-3} Pa) at 850 °C. In this method, the TiC scaffold was preplaced horizontally in the chamber while the ZL107 alloy was stored in a metal tube outside the chamber. After the wetting temperature (850 °C) was reached, the ZL107 alloy was dropped through an open alumina tube and rested on the TiC surface in a solid state. Photographs were then immediately taken to record the melting of the alloy and the movement of the drop during isothermal dwelling. This method offers the advantage of avoiding the pre-interaction of the alloy with the scaffold as well as enabling to the measurement of initial contact angle and wetting and infiltration kinetics. However, because of the coverage of a tenacious oxide film at the Al alloy surface and the porous nature of the TiC scaffold, the intrinsic wettability of TiC by the ZL107 alloy was difficult to be determined. Therefore, a dispensed sessile drop method [23] and a dense TiC substrate were further employed to measure the intrinsic wettability in this system. In the dispensed sessile drop method, the ZL107 alloy was melted and then extruded through a narrow orifice (1 mm in diameter) at the bottom of the alumina tube before it contacted the dense TiC surface. As a consequence, the initial oxide film covering the Al alloy surface was mechanically removed.

After the wetting experiment, contact angles and drop geometric parameters such as base diameter, height and volume were directly measured from the captured drop profiles using an axisymmetric drop shape analysis program. The samples were cut and polished for microstructural observation by using an optical microscope (Axio Imager A2m, Carl Zeiss, Germany), and phase constituents were identified by X-ray microdiffraction (D8 Discover with GADDS, Bruker AXS, Germany) using a 100 μm beam diameter.

2.3. Preparation of the composites and their characterization

The formation of a considerable amount of brittle and hydrolytic Al_4C_3 phase was confirmed by the XRD analysis on the wetting sample. In order to control its formation, we placed a small piece of Al-5 wt% Ti alloy between the TiC scaffold and the ZL107 alloy (the Ti mass concentration was designed to be about 0.3% in the total alloy after mixing). The presence of Ti in the Al melt could greatly reduce the interfacial reaction and simultaneously promote the wettability between the Al alloy and TiC [24]. In addition, we employed gas pressure to accelerate the infiltration process and thus greatly reduced the interaction between the liquid alloy and the TiC scaffold.

Fig. 1 shows the schematic diagram of the gas pressure infiltration apparatus. The TiC scaffold and the Al alloy were placed in an Al_2O_3 crucible, which was then enclosed in a Ni-based superalloy furnace. The furnace was evacuated to a vacuum about 10 Pa and then heated at a rate of 5 °C/min. The molten metal sealed the crucible wall during heating upon to 850 °C. After the temperature was reached, a high-purity (99.999%) Ar gas was then introduced into the furnace and the pressure was controlled at 1 MPa, which was sufficient to drive the liquid metal into the open pores of the TiC scaffold. The pressure was maintained for 1 min and then the furnace was cooled at 5 °C/min.

The microstructures of the composites were observed by using a scanning electron microscope (SEM, Evo18, Carl Zeiss, Germany) equipped with an energy dispersive spectrometer (EDS). Phases were identified by X-ray diffraction (XRD, D/Max 2500PC Rigaku, Japan). The elastic modulus of the composites was measured by using an ultrasonic pulse echo technique, in which the velocities of longitudinal wave and shear wave were determined by an ultrasonic thickness gauge (Olympus 38 DL PLUS, USA) with longitudinal wave probe (M112-RM, 10 MHz) and shear wave probe (V156-RM, 5 MHz) [16]. The compressive strength of the composites was measured by a universal material testing machine (Instron 5689 Corp., USA) using samples of 5 mm × 5 mm × 10 mm under a loading rate of 0.18 mm/min, and three-point bending strength was determined using samples of 3 mm × 4 mm × 20 mm with a span of 18 mm under a rate of 0.5 mm/min, respectively.

3. Results

Fig. 2 shows the typical microstructures of the lamellar TiC scaffolds with different solid loadings after sintering. With the increase in the initial solid loading, both the thickness of the ceramic lamellae and the spacing between two adjacent layers increase. Because of low sintering temperature, the scaffolds showed relatively low strength and the TiC particles were not closely connected. Many cavities could be observed in the ceramic lamellae. Besides, small dendrites with a height of 1–5 μm formed on one side of the lamellae, similar to the nano-asperities

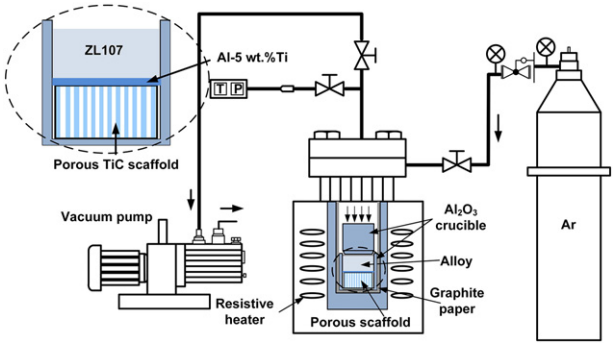

Fig. 1. Schematic diagram of gas pressure infiltration apparatus.