

Short communication

High compressive strength in nacre-inspired Al–7Si–5Cu/Al₂O₃–ZrO₂ composites at room and elevated temperatures by regulating interfacial reaction



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ABSTRACT

Al–7Si–5Cu/Al₂O₃–ZrO₂ composites with nacre-like structures were prepared via ice-templating and gas pressure infiltration techniques. The composites were subsequently heat-treated at 850 °C for 0, 30, 60, 90 and 120 min to regulate the interfacial reaction between Al and ZrO₂. The yield of larger (Al_{1–m}, Si_m)₃Zr and ZrSi₂ phases increased with longer dwell times. The compressive strength initially increased and then decreased. The highest strength was observed in composites treated for 60 min and reached 1600 ± 40, 1261 ± 30 and 1033 ± 22 MPa at temperatures of 20, 150 and 300 °C, respectively. These values increased by 30–40% as compared to those of the non-treated counterparts and were 2-, 5- and 12-fold more than those of the matrix alloy, respectively, which is demonstrative of the material's excellent load-bearing capacity, particularly at elevated temperatures.

1. Introduction

Advances in diverse fields such as aerospace, buildings, transportation, and energy require the development of new, high-performance structural materials. Among them, metal–ceramic composites are ideal candidates because they are lightweight, strong and capable of working at high temperatures [1]. However, few of these applications have yet to reach commercialization, mostly because of the high cost of these materials and difficulties in their processing [1,2]. Besides, these materials are far from meeting the desired performance. One approach in this quest is by mimicking the architecture of natural materials.

Materials scientists have recently pursued the development of biomimetic strategies for artificial nacre materials, garnering inspiration from the distinctive brick-and-mortar structure and properties exhibited in nacre. For example, Deville et al. [3] advocated the use of an ice template to prepare lamellar scaffolds and discovered that the resulting skeleton had a higher compressive strength as compared to samples of the same porosity prepared by other methods. Subsequently, Launey et al. [1] fabricated Al–Si/Al₂O₃ composites with a nacre-like structure by infiltrating an Al–Si alloy into a lamellar Al₂O₃ scaffold prepared from an ice template under gas pressure, of which the composite fracture toughness and toughening mechanism

were investigated. Roy et al. [4] prepared similar materials by squeeze casting and measured the compressive strength at room temperature. A compressive strength plateau was observed at 688 MPa following composite loading in the lamellar direction with a minimum value of 250 MPa in the perpendicular direction. Furthermore, they thoroughly investigated the elastic moduli [5], internal load transfer mode [6] and damage evolution in the composites [4]. More recently, Liu et al. [7] produced 2024Al/SiC composites by squeeze casting a molten 2024Al alloy into lamellar-structured SiC scaffolds at 80 MPa followed by T6 heat treatment. The resulting 2024Al/10 vol%SiC composites exhibited high flexural strength and fracture toughness (931.3 MPa and 18.8 MPa m^{1/2}, respectively). In our prior work, we also fabricated similar-structured composites (Al–Si–Mg/SiC [8] and Al–Si–Mg/Al₂O₃–ZrO₂ [9]) by way of pressureless infiltration and investigated mechanical properties of the composites. The highest compressive strength reached 952 ± 24 MPa for Al–Si–Mg/SiC and 997 ± 60 MPa for Al–Si–Mg/Al₂O₃–ZrO₂.

The above-mentioned studies are limited to the characterization of mechanical properties of nacre-inspired materials at room temperature. To the best of our knowledge, no study has yet to evaluate material performance at high temperatures. The unique lamellar structure produced from the ice-templating of the metal–ceramic

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composite is believed to be a good load-bearing material at elevated temperatures. Furthermore, the composite interface plays a significant role in controlling the load transfer from the matrix to the reinforcing phase. However, this concern has not been raised in previous studies. Therefore, in the present study, we prepared nacre-like Al–7Si–5Cu/Al₂O₃–ZrO₂ composites, tailored their structures and phases by regulating the interfacial reaction and finally measured their compressive properties at room and elevated temperatures.

2. Experimental procedure

Lamellar Al₂O₃–ZrO₂ scaffolds with an initial solid loading of 30 vol % and an Al₂O₃/ZrO₂ weight ratio of 3:7 were prepared by ice-templating (also named freeze casting) at –20 °C and then sintered at 1450 °C for 2 h [10]. The porous ceramic scaffolds were 30 mm in diameter and 25 mm in height. They were subsequently infiltrated with a commercial alloy (ZL107) at 850 °C under a 2 MPa Ar gas. The main composition of ZL107 was Al–7Si–5Cu. The composites were then heat-treated at a temperature of 850 °C for 0, 30, 60, 90 and 120 min to regulate the interfacial reaction between the Al alloy and ZrO₂. The heating and cooling rates were controlled at 10 °C/min.

The composite microstructures were observed using a scanning electron microscope (SEM, Evo18, Carl Zeiss, Germany) equipped with an energy dispersive spectrometer (EDS). The phases were identified by X-ray diffraction (XRD, D/Max 2500PC Rigaku, Japan). The composites were cut into dimensions of 5 mm×5 mm×10 mm from the upper and lower parts, respectively. The compressive strength was then measured along the lamellar direction at 20, 150 or 300 °C using a universal material testing machine (Instron 5689, Instron Corp., USA)

at a loading rate of 0.18 mm/min. The final value was the average of three measurements.

3. Results

Fig. 1 presents the microstructures in the middle of the upper and lower parts of the composites along the lamellar direction following heat treatment at 850 °C for 0, 60 or 120 min. The alloy (black) completely infiltrated the porous ceramic scaffold and formed a nacre-like lamellar structure. The thickness of the lamellae in the lower part is significantly smaller than that in the upper part due to the different aqueous ceramic slurry solidification velocities as well as the different ice growth rates during freeze casting [10]. In addition, no visible reaction product was observed in the composite without heat treatment (i.e., t=0 min). However, at longer times, the products were detected in the metal layers.

The EDS analysis indicated (Al_{1–m}, Si_m)₃Zr phase (hereafter denoted as τ_1) to be the primary reaction product. At 850 °C, ZrO₂ reacted with Al to form Al₂O₃ and Zr. The Zr atoms then diffused into the alloy layer and combined with Al to form Al₃Zr. The reaction can be expressed as:



Because Al and Si atoms have similar atomic radii, Al can be partly substituted by Si in the Al₃Zr crystal, which develops into the τ_1 phase. In some Si-rich regions, the formation of needle-like ZrSi₂ phase is more thermodynamically favorable [11] (see Fig. 1(b–c)). The quantity and size of the τ_1 and ZrSi₂ phases increased considerably at longer reaction time, which was confirmed by the SEM observations and XRD analysis (Fig. 2). The phases further developed into “bridges” between

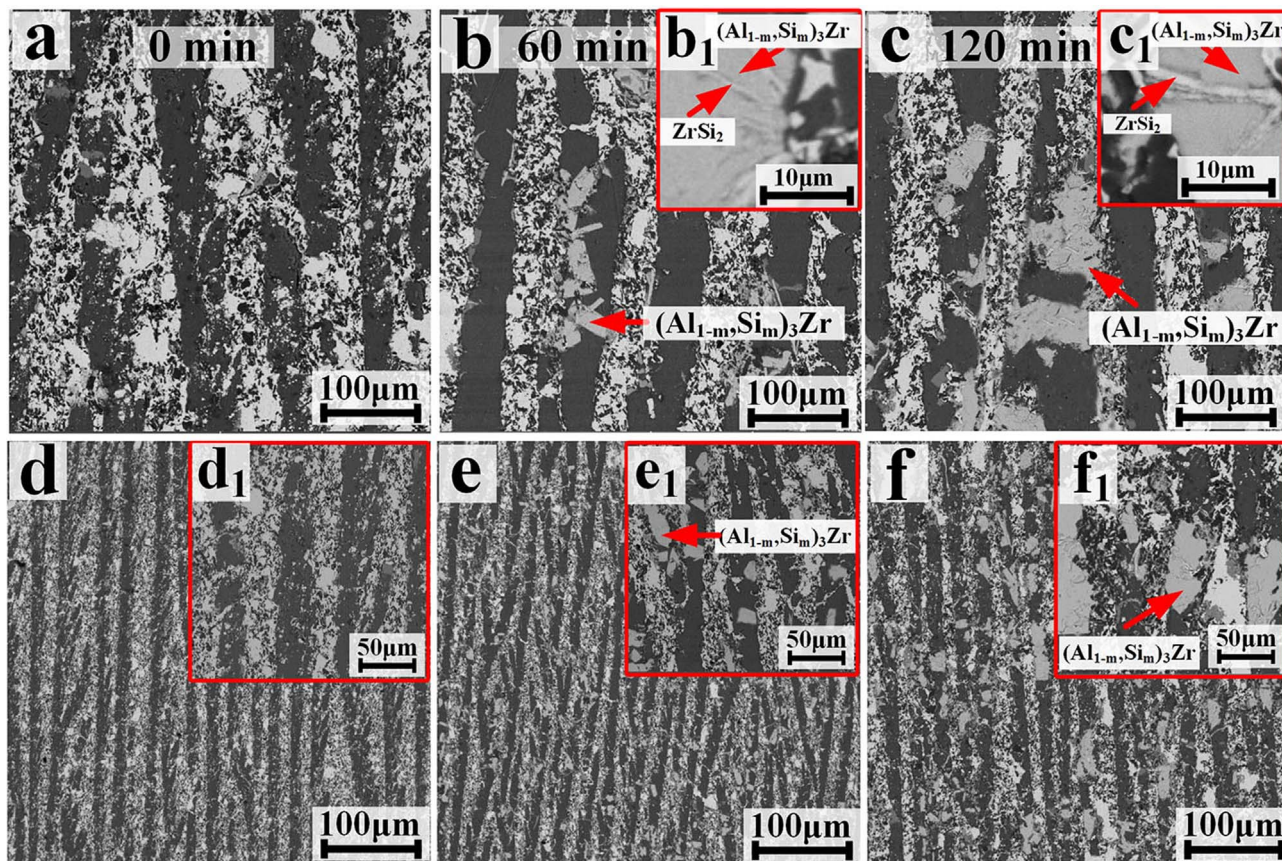


Fig. 1. Backscattered electron images of the composite microstructures following heat-treatment at 850 °C for different times: (a, d) 0 min; (b, e) 60 min; (c, f) 120 min. Images (a–c) correspond to the middle sections of the upper part and (d–f) to the middle of the lower part.

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