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Effect of pyrolytic carbon interface thickness on microstructure and mechanical properties of lightweight zirconium boride modified carbon-bonded carbon fiber composites

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ABSTRACT

To improve the mechanical properties of carbon-bonded carbon fiber (CBCF) composites, they are firstly fabricated by chemical vapor deposited (CVD) pyrolytic carbon (PyC) coating layer on the carbon fiber surface, and then modified by zirconium boride (ZrB_2) using three cycles of precursor infiltration and pyrolysis (PIP) process. The effects of different PyC interface thickness on the microstructure and mechanical properties of ZrB_2 modified PyC coated CBCF (PyC–CBCF/ZrB_2) composites were studied and characterized. As the PyC thickness increased from 0.5 to 3.6 μm, the flexural properties of PyC–CBCF/ZrB_2 composites are noticeably enhanced in *x*/*y* and *z* direction, respectively. Mechanical enhancements for PyC–CBCF/ZrB_2 composites are mainly attributed to the effective interface bonding between carbon fiber and PyC, crack deflection and branching within the laminar PyC layer and carbon fiber pullout from PyC interface coating.

1. Introduction

Carbon-bonded carbon fiber (CBCF) composites possess many excellent properties such as low density, low thermal conductivity, low coefficient of thermal expansion (CTE), good resistance of irradiation and high temperature capability [1–4]. Although the deposition of external coatings on CBCF composites can prevent them from oxidation in oxidation-containing atmospheres, CBCF composites have a poor mechanical strength to meet the increasing demand of actual application [2]. Therefore, the enhancement of mechanical properties is crucial for CBCF composites used in high temperature and load-bearing environment.

To overcome these limits, many researchers have focused on the surface modification of carbon fibers to enhance the interfacial adhesion between carbon fibers and matrix. For example, Li et al. [3] studied the C/C composites with in situ grown CNFs using natural gas as carbon source. The CNFs are beneficial to the improvement of mechanical properties of C/C composites due to bridging effect which redistributes the load while the cracks extend toward fibers or CNFs leading to higher toughness as the more complex crack propagation channels dissipate more energy. Lim et al. [4] succeeded to prepare C/C composites with improved mechanical strength by using a precursor made of CNFs grafted on carbon microfilaments. The as-synthesized carbon nanofibers acted as a “solid glue” through the formation of numerous nano-and micro junctions between the microfilaments and exhibited high elasticity and strength compared to raw materials. Another method is to invite the preparation process on the mechanical properties of C/C composites carried out by Weisshaus et al. [5]. The result indicated that the duration of graphitization tended to reduce compression strength of C/C composites. It is generally accepted that effective interfacial carbon layers can be remarkable enhancement of mechanical properties for C/C composites due to good thermal coefficient matching and remarkable mechanical property of interfacial carbon layers [6–9]. Especially for porous carbon-based composites, it can form a uniform thinner carbon layer on the carbon fiber surface by chemical vapor deposition (CVD), in which the pores provide the diffusion channel for gas containing carbon in a CVD process.

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In this work, pyrolytic carbon (PyC) modified porous CBCFs (PyC–CBCF) composites were fabricated by the formation of a PyC coating layer on the carbon fiber surface via a CVD method. And then, the lightweight ZrB_2-modified PyC–CBCF (PyC–CBCF/ZrB_2) composites were fabricated by polymeric precursor infiltration and pyrolysis (PIP). The main purpose of the paper is to investigate the effect of PyC interphase thickness on the mechanical strength and fracture behavior of porous PyC–CBCF/ZrB_2 composites.

2. Experimental

The schematic illustration of the typical microstructure of CBCF composites and manufacturing process for PyC–CBCF/ZrB_2 composites are shown in Fig. 1. As seen from Fig. 1(a)–(c), the CBCF composites consist of chopped carbon fiber network bonded together at the intersections of the fibers by discrete regions of vitreous carbon. In addition, the discontinuous carbon fibers are orientated into layers to form a 2D planar random structure at *xy* direction [10,11]. The schematic illustration of manufacturing process for PyC–CBCF/ZrB_2 composites is shown in Fig. 1(d)–(g). Firstly, porous PyC–CBCF composites were fabricated by the formation of a PyC coating layer on the carbon fiber surface via a CVD method (Fig. 1(d), (e)). And then, the lightweight PyC–CBCF/ZrB_2 composites were fabricated via a PIP process (Fig. 1(f), (g)).

2.1. Starting materials

Rayon-based carbon fiber (diameter of 75 μm and length of 1 mm) was purchased from Ji lin Ji yan High-tech Fibers Co., Ltd. (Jilin, China). Phenolic resin powder (PF4090, fineness >95%/200 mesh) was obtained from the Holyspring chemical Co., Ltd. (Juxian, China). Polyethylene imine (PEI) with an average molecular weight of 10,000 was obtained from Aladdin Co. Ltd. (Shanghai, China). ZrB_2-containing precursors was obtained from Institute of Process Engineering (Chinese Academy of Science, Beijing, China).

2.2. Preparation of CBCF composites

CBCF composites were prepared using the classical pressure filtration procedure [12]. A 200 ml aqueous solution consisting of 60 mg PEI was subject to a vigorous stirring in a 250 ml beaker, and 5 g short carbon fiber was then added slowly to the solution to form a water-based slurry. After stirring for 20 min, the same weight of phenolic resin powder was added to the slurry and the slurry was stirred for another 30 min to achieve good dispersion of water-based slurry. The water-based slurry was then poured into a cylindrical mould [13] to remove the water from the slurry under an appropriate pressure to obtain a desired density. The acquired wet billets were cured at 80 °C for 2 h and 150 °C for 3 h. Then the cured samples were carbonized at 1000 °C in Ar for 1 h. The CBCF composites with a density of 0.26 g/cm^3 and porosity of 84.71% were prepared.

2.3. Preparation of PyC–CBCF–ZrB_2 composites

PyC was deposited on the fibers surface in CBCF framework by chemical vapor deposition (CVD) at 1200 °C using propane as carbon source. Three different PyC interface thicknesses of 0.5, 2.6 and 3.6 μm were obtained by controlling deposition time for 30, 100 and 150 h, respectively. Ultimately, PyC–CBCF composites with different interface thicknesses were impregnated into ZrB_2 polymeric precursor to prepare ZrB_2 modified PyC–CBCF composites by a PIP method [2]. In a typical experiment, the PyC–CBCF composites were impregnated into ZrB_2-containing polymeric precursor using a vacuum infiltration devise, drying at 80 °C in a drying oven at Ar atmosphere. The pyrolysis was performed at a heating rate of 2 °C/min to the desired temperature of 1500 °C for 2 h under flowing Ar atmosphere. The composites were densification by another two cycles of ZrB_2-containing polymeric precursor infiltration and pyrolysis.

For comparison, CBCF composites without CVD PyC were fabricated by three cycles of ZrB_2-containing polymeric precursor infiltration and pyrolysis under the same condition.

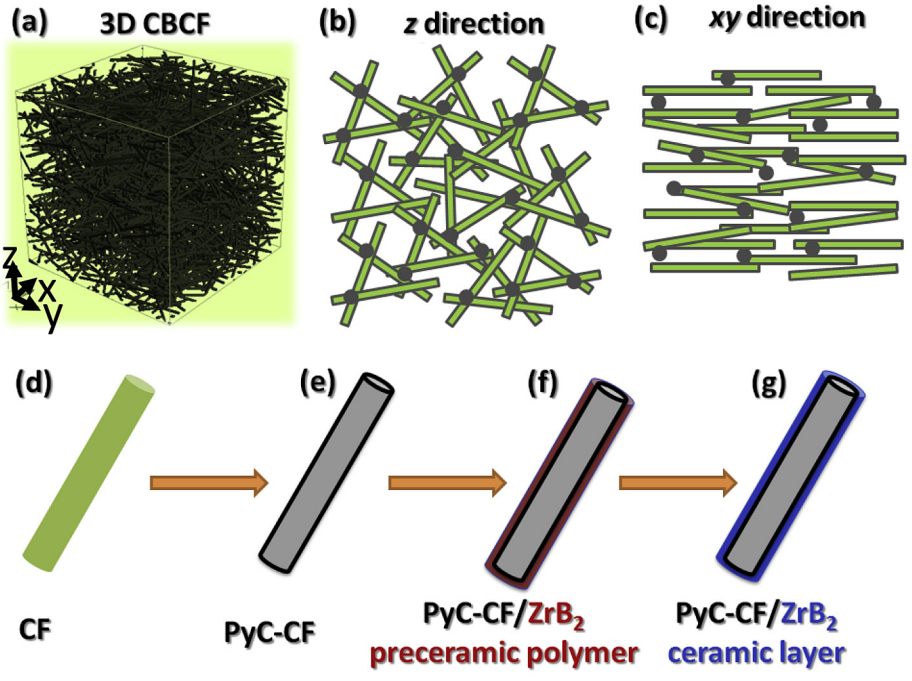

Fig. 1. Schematic illustration of the typical microstructure of CBCF composites and the manufacturing process for PyC–CBCF/ZrB_2 composites.