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Mechanical properties and fracture behaviors of C/C composites with PyC/TaC/PyC, PyC/SiC/TaC/PyC multi-interlayers

Xiang Xiong*, Ya-lei Wang, Zhao-ke Chen*, Guo-dong Li

State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, China

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ABSTRACT

Carbon/carbon (C/C) composites with PyC/TaC/PyC or PyC/SiC/TaC/PyC multi-interlayers were prepared by isothermal chemical vapor infiltration, followed by Furan resin impregnation and carbonization. Microstructures, mechanical properties including flexural strength, ductile displacement, and fracture behaviors of composites were studied. Furthermore, composites were heat treated at 2000 °C to study the effects of heat treatment on mechanical properties and fracture behaviors. PyC/TaC/PyC and PyC/SiC/TaC/PyC multi-interlayers have been deposited uniformly in C/C composites. With the introduction of PyC/TaC/PyC multi-interlayers in C/C composites, the flexural strength decreases; however, the ductile displacement increases. The fracture behavior changes from brittleness (0% TaC) to pseudo-ductility (5% TaC) and high toughness (10% TaC). When PyC/SiC/TaC/PyC multi-interlayers are introduced in C/C composites, the flexural strength is improved remarkably from 270 MPa to 522 MPa, but the ductile displacement decreases obviously from 0.49 mm to 0.24 mm, and the fracture behavior becomes brittle again. After heat treatment at 2000 °C, the flexural strength decreases, but the ductile displacement increases and pseudo-ductility or high toughness can be obtained.

1. Introduction

Carbon/carbon (C/C) composites are promising candidates for many applications, particularly as aerospace and aircraft high-temperature structures [1,2]. Nevertheless, a critical drawback of C/C composites is the poor oxidation resistance, which limits their long time applications in high-temperature oxidizing environment [3]. The main methods to improve the oxidation-resistance properties of C/C composites are doping and coating [4–7]. However, the coating method has a disadvantage of cracks in the protective coating due to the mismatch of coefficient of thermal expansion (CTE) between the substrate and the coating materials, while the doping technique can only provide protection from oxidation with a very limited effect.

Labruquère and coworkers [8,9] had proposed an internal-protection method to improve the oxidation-resistance properties of C/C composites. They coated B–C, Si–B–C and Si–C phases on carbon fibers as interlayers in C/C composites. Their results had showed that the oxidation resistance of C/C composites can be improved remarkably with the introduction of the above interlayers. However, these C/C composites with B–C, Si–B–C and Si–C interlayers cannot be applied in ultra-high temperatures due to the gasification of the glassy phase.

The refractory carbides, such as HfC, TaC, ZrC and NbC, are promising materials to improve the oxidation resistance of C/C composites in ultra-high-temperature environments (above 1700 °C) [10]. Our previous works [11,12] had introduced some PyC/TaC/PyC and PyC/SiC/TaC/PyC multi-interlayers in C/C composites. However, the previous works have not described the mechanical properties and fracture behaviors of these composites.

The mechanical properties and fracture behaviors of fiber reinforced ceramic matrix comosites are sensitive to the bonding strength at the fiber/matrix interfaces [13]. Strong interfacial bonding usually results in high strength but brittle fracture of the composites, while too weak bonding causes the fibers to be pulled out of the matrix at a relatively low load [14,15]. Undoubtedly, the PyC/TaC/PyC and PyC/SiC/TaC/PyC multi-interlayers will influence the interfacial bonding strength, and then the mechanical properties and fracture behaviors of the composites.

In this paper, PyC, SiC, TaC phases were deposited in C/C composites using chemical vapor infiltration (CVI). The effects of the PyC/TaC/PyC and PyC/SiC/TaC/PyC multi-interlayers on mechanical properties and fracture behaviors were studied. In addition, the composites were heat treated to study the effects of heat treatment on mechanical properties and fracture behaviors.

* Corresponding authors. Tel./fax: +86 731 8836079.
E-mail address: pallettechen@163.com (Z.-k. Chen).

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2. Experimental

2.1. Preparation

Needle-integrated felts with an original density of 0.56 g/cm^3 were used as preforms. The carbon fiber was PAN-based (T300, 12k, Toray, Japan). The felts were prepared by the three-dimensional needling technique, starting with repeatedly overlapping the layers of 0° non-woven fiber cloth, short-cut-fiber web, and 90° non-woven fiber cloth with needle-punching step by step. A multi-functional isothermal CVI system, as shown in Fig. 1, was used to deposit PyC, SiC and TaC phases.

Ar and C_3H_6 were used to deposit PyC phase by isothermal CVI at 900–1150 °C and pressure less than 2 kPa. Methyltrichlorosilane (MTS, CH_3SiCl_3) and H_2 were used to deposit SiC phase. MTS was vaporized at 34.5 °C in evaporator and carried by bubbling hydrogen. The conditions for deposition of SiC phase were as follows: 1100 °C, pressure less than 2 kPa, and the molar ratio of H_2 to MTS 10. $TaCl_5$–C_3H_6–Ar mixtures were used to deposit TaC phase at 800 °C and 200–600 Pa [16]. Then preforms were further deposited with PyC phase (by CVI) and densified with resin carbon (by impregnation and carbonization with 2–3 circles). The impregnation and carbonization consisted of three steps: Furan resin impregnation in preforms, solidification at 180–200 °C for 1 h, and carbonization at 800–1000 °C for 2 h. In addition, some specimens were treated at 2000 °C for 2 h.

2.2. Characterization

Samples with a size of 40 mm × 5 mm × 3 mm were used to test the flexural strength by INSTRON CSS-44100 machine. The gauge size was 30 mm and the crosshead speed was 0.5 mm/min. The load–displacement curves were recorded by computer at the same time. The flexural strength of the composites was estimated according to the following equation [17],

$$\sigma_f = \frac{3PL}{2bh^2}; \quad (1)$$

where σ_f is the flexural strength, MPa; P the maximum load, N; L the gauge size, mm; b and h are the width and thickness of samples, mm.

The fracture behavior of fiber reinforced ceramic matrix composites will undergo four processes: elastic strain, matrix crack, fiber pull-out and complete failure. At matrix crack and fiber pull-out stages, the pseudo-ductile deformation may occur. In this paper, ductile displacement is presented to characterize the pseudo-ductile deformation of the composites. In the load–displacement curve, a line (m) parallel to the elastic strain curve (n) is drawn through the peak load point. The intercept values of the two parallel lines with the displacement axis are used to calculate the ductile displacement,

$$\Delta D = D_m - D_n; \quad (2)$$

where ΔD is the ductile displacement, mm; D_m, D_n the intercept values of line m and line n with the displacement axis respectively, mm.

Microstructures and fractographs of composites were analyzed by a JEOL-6360LV scanning electron microscope (SEM), at a working voltage of 25 kV. The element composition of multi-interlayers in composites were analyzed by an energy spectrum analysis.

3. Results and discussion

3.1. Microstructures

Fig. 2 shows the microstructures of C/C composites with PyC/TaC/PyC and PyC/SiC/TaC/PyC multi-interlayers.

As can be seen from Fig. 2(a) and (b), two different multi-interlayers have been deposited uniformly in C/C composites respectively. Resin carbon fills up the spaces between the tows. Fiber bridging occurs, i.e. fibers are physically in contact or the distance between fibers is less than twice the coating thickness. In addition, some delaminations between the matrix and the multi-interlayers can be observed. The delamination results from the volume shrinkage of resin carbon in carbonization.

Fig. 2(c) and (d) shows the structures of the two different multi-interlayers. Fig. 2(e) and (f)shows the composition analyses of points M and N in Fig. 2(d). From the microstructure and composition analysis, we can know that, the first layer is pure PyC with a thickness of about 0.2–0.5 μm (to achieve a good bonding with the fiber [15]), and the following layers are TaC and PyC, or SiC, TaC and PyC. Both SiC and TaC interlayers have a thickness of about 1 μm. The combination of C_f, PyC, SiC, TaC and PyC interlayers is compact, with no crack in.

3.2. Mechanical properties

Table 1 lists the mechanical properties and fracture behaviors of the composites with different multi-interlayers.

The typical load–displacement curves of C/C composites with multi-interlayers are shown in Fig. 3.

C/C composites without multi-interlayers fail in a brittle fashion (Fig. 3(a)) while C/C composites with PyC/5% TaC/PyC multi-interlayers show 'elastic–plastic' manners. In Fig. 3(b), the load–displacement curve can be divided into three segments: linear rise (OP), non-linear rise (PQ) and flat displacement (QR), corresponding to three stages: matrix elastic deformation, matrix crack and fiber (or multi-interlayer) pull-out respectively. When the displacement reaches the maximum value (point R), the composites fail completely. The failure of C/C composites with PyC/10% TaC/PyC multi-interlayers occurs in a controlled manner (Fig. 3(c)). The load–displacement curve shows elastic response in the initial stage

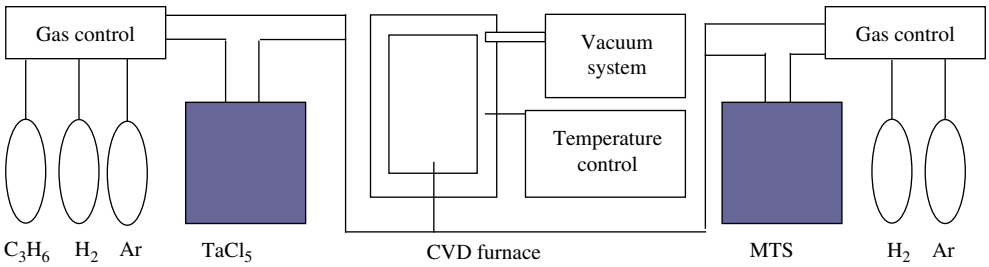

Fig. 1. Schematic drawing of a multi-functional isothermal CVI system.