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Microstructure and mechanical properties of carbon fiber reinforced multilayered $(PyC–SiC)_n$ matrix composites

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ABSTRACT

Carbon fiber reinforced multilayered $(PyC–SiC)_n$ matrix $(C/(PyC–SiC)_n)$ composites were prepared by isothermal chemical vapor infiltration. The phase compositions, microstructures and mechanical properties of the composites were investigated. The results show that the multilayered matrix consists of alternate layers of PyC and β-SiC deposited on carbon fibers. The flexural strength and toughness of $C/(PyC–SiC)_n$ composites with a density of 1.43 g/cm^3 are 204.4 MPa and 3028 kJ/m^3 respectively, which are 63.4% and 133.3% higher than those of carbon/carbon composites with a density of 1.75 g/cm^3. The enhanced mechanical properties of $C/(PyC–SiC)_n$ composites are attributed to the presence of multilayered $(PyC–SiC)_n$ matrix. Cracks deflect and propagate at both fiber/matrix and PyC–SiC interfaces resulting in a step-like fracture mode, which is conducive to fracture energy dissipation. These results demonstrate that the $C/(PyC–SiC)_n$ composite is a promising structural material with low density and high flexural strength and toughness.

1. Introduction

Carbon/carbon (C/C) composites have been widely studied due to their excellent advantages, such as low density, high specific strength, low thermal expansion coefficient and superior ablation resistance [1–3]. These properties make them attractive in aeronautical and astronautical applications as structural materials. Especially in the high-technology field of solid rocket propulsion, C/C composites have the advantage of excellent ratio of mechanical properties versus density at elevated temperature, which can satisfy the need of a high thrust-to-mass ratio, i.e., high thrust with a low engine mass [4]. A further development is to lighten the weight as well as maintain or improve the strength of materials. Besides, low flexural toughness and brittle fracture behavior of C/C composites have seriously limited their additional usage [5]. Therefore, challenges and concerns should be focused on these weak points.

It has been assumed that the best interphase materials might be those with a layered crystal structure (pyrocarbon (PyC), hexagonal-BN) or an alternate layered microstructure ($(PyC–SiC)_n$, $(BN–SiC)_n$) [6,7]. Most investigations show that the multilayered interphases are beneficial to improving mechanical properties, oxidation resistance and neutron irradiation tolerance of composites [7–12]. Furthermore, the concept of multilayered structure has been extended to the matrix itself yielding laminated matrix composites (LMCs) [13–15], with a view to improving the performance of brittle materials, such as C/C composites.

Up to now, enormous efforts have been exerted on LMCs. Appiah et al. [14,15] studied the effect of deposition temperature gradient on microstructural differences of laminated PyC–SiC matrix composites fabricated by forced flow-thermal gradient chemical vapor infiltration (F-CVI), and characterized the detailed interfacial microstructures between the fiber and matrix and the PyC–SiC interfaces within the matrix. Lamouroux et al. [16] introduced a multilayer SiC–B–C ceramic matrix to a 2.5D preform by pulsed CVI (P–CVI) and found that the multilayered ceramic matrix could improve the oxidation resistance and thermomechanical behavior of the composites. Recently, Yang et al. [17] prepared a 3D $(BC_x–SiC)_n$ multilayered matrix composite reinforced by carbon fiber, and the composite exhibited a remarkable metal-like yield stage and high flexural strength. Xie et al. [18] showed that the laminated structure of SiC_w–SiC ceramic composites can improve the flexural strength, tensile strength and fracture toughness through whiskers pullout, cracks deflection and bridging between interfacial and inter-laminar zones. In our previous work [19], it was found that C/C composites with multilayer-textured PyC matrix owned a relatively high flexural strength and pseudo-plastic fracture behavior. Such studies have demonstrated that LMCs present strength and toughness improvements by combining the benefits of laminated structures with the fiber reinforcement. Besides, the properties of LMCs in view of a given application can be optimized through materials design at the level of the components (fiber and matrix).

Given the fact that both LMCs and multilayered $(PyC–SiC)_n$ interphases have excellent advantages in the improvement of mechanical properties, the introduction of multilayered $(PyC–SiC)_n$ structure as matrix of the composites seems promising. Therefore, the knowledge about the mechanical properties of multilayered $(PyC–SiC)_n$ matrix composites is essential in order to assess their potentially operational performance for aeronautical and astronautical applications.

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To date, the mechanical properties of multilayered $(PyC–SiC)_n$ matrix composites reinforced by carbon fiber have been rarely investigated, especially for the purpose of lightweight design. In the present work, carbon fiber reinforced multilayered $(PyC–SiC)_n$ matrix ($C/(PyC–SiC)_n$) composites were prepared using isothermal CVI (I-CVI). The main purposes were to investigate the matrix microstructure and mechanical properties of the composites, which were compared with those of C/C composites with pure PyC matrix fabricated by I-CVI. Meanwhile, a $C/(PyC–SiC)_n$ composite with low density and high flexural strength and toughness was obtained. The enhancement mechanism of multilayered matrix was also investigated.

2. Experimental

2.1. Raw materials

2D needle-punched integrated felts with a density of 0.45 g/cm^3 were used as preforms. Schematic drawing of the architecture of the preforms is shown in Fig. 1. They were made up of layers of 0° non-woven carbon fiber cloth, short-cut fiber web, 90° non-woven carbon fiber cloth and short-cut fiber web after needle-punching in the vertical direction step by step. The PAN-based carbon fibers (T300, Toray Company, Tokyo, Japan) had a typical density of 1.76 g/cm^3 and an average diameter of 7 μm.

2.2. Preparation process

$C/(PyC–SiC)_n$ composites were prepared from the preforms by deposition of 2 PyC–SiC sequences ($(PyC–SiC)_2$, $n = 2$) using I-CVI. The PyC matrix was deposited with the methane partial pressure of 15 kPa at 1000 °C, and the flow rate of methane was controlled at 400 mL/min. The SiC matrix was deposited from methyltrichlorosilane (MTS, CH_3SiCl_3). MTS vapor was carried into chamber by bubbling hydrogen. Argon was employed as the diluted gas. The conditions for deposition of SiC matrix were as follows: the deposition temperature was 1100 °C, the total pressure was 3 kPa, the molar ratio of H_2 to MTS was 10, and the argon and hydrogen flow rates were 500 and 200 mL/min, respectively. The deposition time for each matrix was sequentially set as 10, 20, 10 and 10 h, respectively. For the purpose of comparison, C/C composites with single PyC matrix were fabricated by the same deposition process of PyC matrix except the deposition time of 120 h.

2.3. Measurements and observation

Three-point bending tests were carried out in accordance with Q/GB 95–92 [20] to evaluate the flexural strength. The two as-prepared composites were cut into rectangular samples with dimensions of 55 mm × 10 mm × 4 mm along the direction paralleled to the non-woven carbon fiber cloth. Samples were randomly chosen to be tested. Before the tests, the densities and open porosities of samples were measured with distilled water by Archimedes method. Tests were performed on a MTS CMT5304-1 kN universal testing machine with a span of 40 mm and a loading speed of 0.5 mm/min. The loading direction was perpendicular to the non-woven cloth. Because of the large scatter of the composites, the number of effective samples was not less than ten for each composite. The toughness of each composite was evaluated through the work of fracture calculated by integration of the nominal flexural stress–strain curves. The results were analyzed by the Weibull distribution method. The Weibull parameters (shape parameter and scale parameter) were calculated according to [21].

The phase compositions of the composites were analyzed by an X-ray diffraction (XRD, DX-2700) device using Cu K_α radiation. The microstructures of the composites were observed on polished cross-sections under a polarized light microscope (PLM, Leica DMLP). The microstructure of multilayered matrix and fracture surface morphology of the composites were observed by scanning electron microscope (SEM, ZEISS SUPRA55) equipped with an energy dispersive spectroscope (EDS, Oxford INCA).

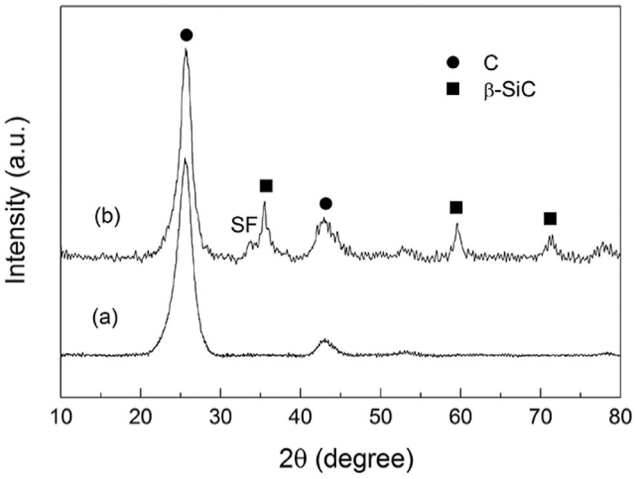

Fig. 2. XRD patterns of (a) C/C composites and (b) $C/(PyC–SiC)_n$ composites.

3. Results and discussion

3.1. Phase compositions

Fig. 2 shows XRD patterns of C/C composites and $C/(PyC–SiC)_n$ composites. It is obvious that only carbon phase occurs in C/C

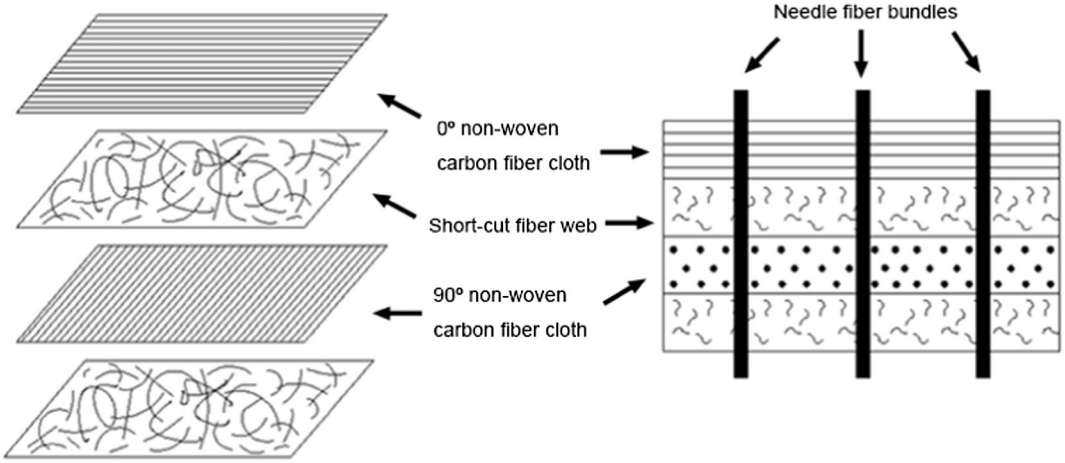

Fig. 1. Schematic drawing of architecture of 2D needle-punched integrated felts.