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Improvement in toughness of carbon/carbon composites using multiple matrixes Hailiang Li^a, Hejun Li^{a,*}, Jinhua Lu^a, Can Sun^a, Yongjie Wang^a, Dongjia Yao^a, Kezhi Li^a, Haipeng Wang^b

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ABSTRACT

Carbon/carbon (C/C) composites with multiple matrixes were fabricated by impregnation/carbonization and chemical vapor infiltration (CVI) using 2-dimensional (2D) carbon felts preform. In order to study the effects of microstructure on mechanical properties, C/C composites with single matrix of pitch based carbon were prepared for comparison. The mechanical properties were tested on CMT5304-30KN universal testing machine. Polarization microscope and scanning electron microscope were used to investigate the microstructures and fracture surface of C/C composites. It was resulted that the flexural strength of C/C composites with multiple matrixes was improved by 83% compared with that of C/C composites with single matrix. Meanwhile, better toughness and pseudo-plastic fracture behaviors were also obtained with multiple matrixes. Multi-layer interfaces between different constituents were formed in the composites which supplied variety of extension paths for cracks. This microstructure character improved the mechanical properties of C/C composites, especially the toughness, as more energy could be dissipated during cracks spread along multiple paths, such as the interface of different components or among the matrixes.

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1. Introduction

Widespread applications of carbon/carbon (C/C) composites in different fields have been developed, attributed to their superior characteristics [1–3], such as low density, high strength, high thermal conductivity, excellent ablation property and low thermal expansion coefficient together with good frictional performance in inert atmosphere.

Nevertheless, low toughness and brittle behaviors, drawbacks of the material have limited its more extensive usage as structural material. Much effort has been done to resolve this problem [4–8]. Proper modification of microstructures of *C/C* composites may improve the mechanical properties as they are mainly controlled by the microstructures. The fracture behaviors of *C/C* composites are mainly determined by the propagation of cracks within the matrix, so by controlling the structure of the matrix, *C/C* composites may have higher mechanical properties, especially better toughness. Gajiwala developed a new approach which incorporated a lamina-by-lamina curing scheme using polyimide resin. It introduced a hybrid matrix of polyimide and phenolic resin that enabled improvement of mechanical properties of the composites [9]. Gao et al. revealed that the introduced Zr could improve the interface bonding and thus enhanced the mechanical properties of *C/C* com-

posites [10]. Xiong manufactured C/C composites with PyC/TaC/PyC multiple interlayers by isothermal chemical vapor infiltration (CVI) and carbonization of furan resin. It was concluded that with introduction of the multiple interlayers in C/C composites, the ductile displacement was increased. The fracture behavior changed from brittleness to pseudo-ductility and high toughness was obtained [11]. Ayatollahi et al. demonstrated that the addition of carbon nanotubes (CNTs) was beneficial to the improvement of toughness of composites. The presence of CNTs had a great effect on enhancement of fracture toughness of the nano-composites [12]. Lin and co-workers [13] reported a toughening concept based on dissolvable phenoxy fibers, which are added at the interlaminar region in a carbon fiber/epoxy composite. The fracture toughness was increased by the added phenoxy fiber. Xiao prepared CNT reinforced C/C composites by in situ grown in unidirectional carbon cloths. The results showed that the existence of CNTs increased the number of interfaces and form the complicated interface in C/C composites which led to higher flexural properties [14]. The studies of Taguchi et al. [15] demonstrated that both flexural and tensile strength of SiC/SiC composites with SiC/C multilayer were approximately 10% higher than composites fabricated without SiC/C layer.

In the above studies, several methods have been reported to improve the mechanical properties of *C/C* composites or *C/SiC* composites. Introduction of multiple layers into composites has been proved to be a useful way to the reinforcement of composites, especially the toughness [16,17]. Nevertheless, toughening of *C/C* composites by multilayer microstructure has been scarcely

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Table 1The properties of mesophase pitch.

Material	Softening point (K)	Volume density (g cm ⁻³)	Mesophase content (%)	C/H mol ratio
Mesophase pitch	548-568	0.65	100	1.56-1.72

studied. So matrix modification was applied as means of toughness enhancement of C/C composites in this work. C/C composites with multiple carbon matrixes were fabricated by CVI and impregnation/carbonization to improve the toughness of composites. The microstructures and mechanical properties of the C/C composites with multiple matrixes were studied and the mechanism of the enhancement of C/C composites was investigated.

2. Experimental

2.1. Materials

The preform materials used in this work were 2D carbon felts with a density of $0.4\,\mathrm{g/cm^3}$ which were fabricated by repeatedly overlapping the layers of 0° weftless ply, short-cut fiber web and 90° weftless ply with needle-punching step-by-step. The mesophase pitch used was supplied by Mitsubishi Gas Chemical, Japan, the properties were showed in Table 1.

2.2. Sample preparation

C/C composites with multiple matrixes were fabricated from the preform by multi-step process. First a layer of pyrolytic carbon was deposited by CVI in an electric resistance furnace with methane as precursor and nitrogen as diluted gas. After the pre-deposition of pyrolytic carbon, the as-obtained samples were pressure-infiltrated with mesophase pitch. Following pressurized mesophase pitch impregnation, the samples were oxidized at atmosphere and then carbonized. The details of impregnation/carbonization can be seen in the former work [18]. After four cycles of impregnation/carbonization, the samples were densified by CVI process for 20 h using an electric resistance furnace with methane as precursor and nitrogen as diluted gas. For comparison, C/C composites with single matrix of pitch based carbon were fabricated by cycles of impregnation/carbonization. C/C composites with multiple matrixes were marked as CC-M and C/C composites with single matrix were marked as CC-S.

2.3. Mechanical property tests

Three-point bending property was tested on CMT5304-30KN universal testing machine, conducted at loading speed of 0.5 mm/min and support span of 40 mm length. The size of

specimens was 55 mm \times 10 mm \times 4 mm. The number of specimens was not less than five for every test point. The flexural strength and modulus were calculated according to the formulas listed below.

$$\sigma_f = \frac{3PS}{2bh^2} \tag{1}$$

$$E_f = \frac{\Delta P_f S^3}{4bh^3 \Delta f} \tag{2}$$

where σ_f is flexural strength, E_f is flexural modulus, P is the maximum of load, S is span, b is the width of specimen, h is the height of specimen, and $\Delta P_f/\Delta f$ is the slope of load–displacement curve.

2.4. Microscopic morphology

The samples were mounted in epoxy resin and successively ground on four grades of silicon carbide paper (400, 600, 800 and 1200 grits) in turns and polished with diamond paste on polishing cloth. Then Leica DMLP optical microscope with plane polarized reflected light was used to produce characteristic interference colors. These interference colors are directly related to the structure of the carbons, particularly the orientation of basal planes. Higher resolution image was obtained using a JSM6460 and 6700 scanning electron microscope (SEM). SEM has allowed observation of fracture surface, interface between the fiber and matrix and morphology of each constituent.

3. Results and discussion

3.1. The microstructure of C/C composite with multiple matrixes

Figs. 1 and 2 show the polarized light micrographs (PLM) of C/C composites with types of matrix microstructures attributed to the multiple steps of fabrication. Two different matrixes of pyrolytic carbon and pitch based carbon were obtained in the composites. Among fiber bundle, the first deposited pyrolytic carbon develops around carbon fiber, the thickness of which is about 2–3 μm . It is observed that carbon fibers are embedded in pyrolytic carbon. After the impregnation/carbonization technique, samples are densified by CVI secondly for 20 h, and from Fig. 2a, it is deduced that the pyrolytic carbon obtained in this process is distributed among the large voids generated after carbonization of mesophase pitch. So the large voids can be filled after the second CVI process,

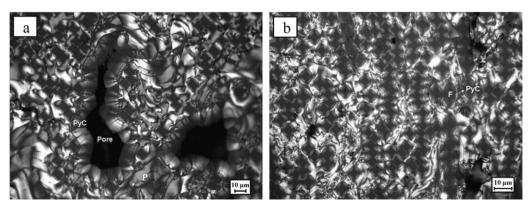


Fig. 1. The PLM of C/C composites with different matrixes (P: pitch based carbon; PyC: pyrolytic carbon).

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