

## Rapid communication

## The reinforcement and toughening of pyrocarbon-based carbon/carbon composite by controlling carbon nanotube growth position in carbon felt

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## ABSTRACT

The flexural properties of pyrocarbon-based carbon/carbon (C/C) composites reinforced by in situ grown carbon nanotubes are reported. The effect of nanotube growth position (on carbon fibers or in the spaces between them) was investigated. A method of reinforcing and toughening C/C composites is proposed and the corresponding mechanisms are discussed.

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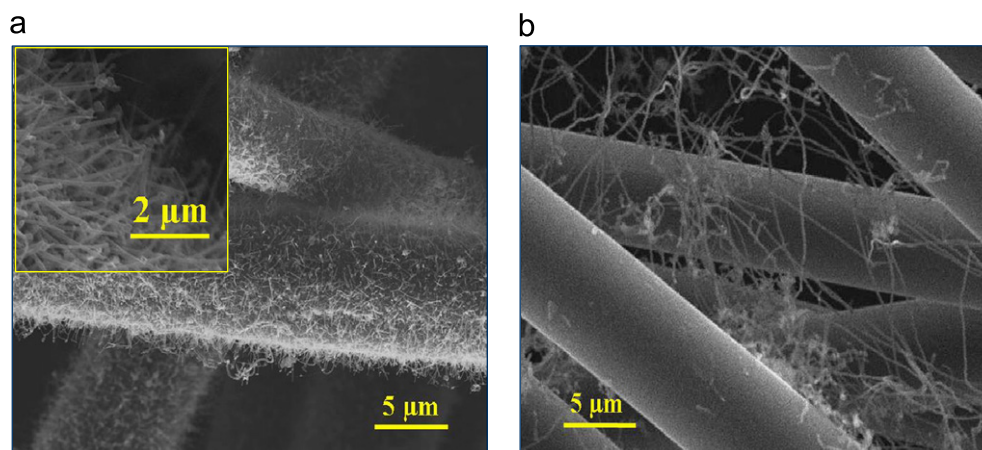


Fig. 1. SEM images of CNTs with different growth positions: (a) CNTs grown on carbon fibers and (b) CNTs grown in the spaces between fibers.

## 1. Introduction

Over the past few decades, much different effort has been devoted to improve the toughness of carbon/carbon (C/C) composites [1–3]. An efficient work is adding nano-filler, such as carbon nanofiber (CNF), into the composites [2,3]. In the cases, resin or pitch carbon was used as the matrix and the toughening mechanisms have been found to be “CNF bridging” and “CNF pull-out”. However, for C/C

composites with pyrocarbon (PyC) matrix, the existing reports show that adding nano-fillers such as carbon nanotube (CNT) always leads to the brittle fracture of the composites despite a largely increased mechanical strength [4–6]. The reasons for the brittle fracture are various. One of them is the failure of the toughening mechanisms mentioned above because the bonding between CNT and PyC is very strong [7] that inhibits the long-distance pull-out of CNT from carbon matrix. Another important one is the overly-enhanced F/M interface bonding because of the introduction of CNTs onto carbon fibers which blocks the fiber pull-out and decreases the fracture toughness [5,6], even the CNT content is very low [5].

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Recently, it is found that carbon matrix with multilayer microstructures is beneficial to the mechanical properties of C/C composites, especially the toughness [8,9]. On the other hand, introducing

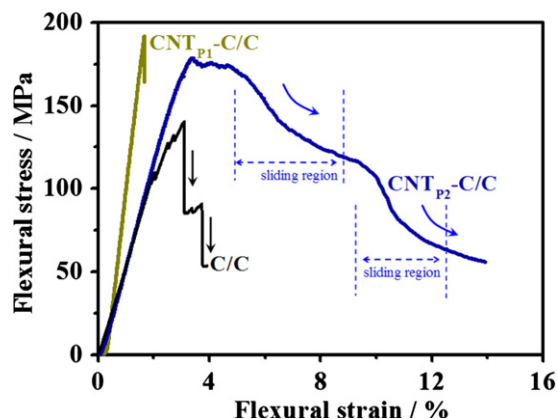


Fig. 2. Representative flexural stress–strain curves of the three composites.

**Table 1**  
Mechanical properties of the three kinds of C/C composites.

| Composites             | CNT content (wt.%) | CNT length( $\mu\text{m}$ ) | Bulk density ( $\text{g} \cdot \text{m}^{-3}$ ) | Flexure strength (MPa) | $F_D$           |
|------------------------|--------------------|-----------------------------|-------------------------------------------------|------------------------|-----------------|
| C/C                    | 0                  | 0                           | 1.55                                            | $143 \pm 5$            | $0.27 \pm 0.07$ |
| CNT <sub>P1</sub> -C/C | 2.0                | 2–4                         | 1.57                                            | $182 \pm 8$            | $0.03 \pm 0.01$ |
|                        | 9.5                | 4–20                        | 1.56                                            | $251 \pm 7$            | $< 0.01$        |
| CNT <sub>P2</sub> -C/C | 1.9                | 8–20                        | 1.57                                            | $179 \pm 10$           | $0.56 \pm 0.10$ |
|                        | 6.2                | 5–18                        | 1.57                                            | $196 \pm 12$           | $0.58 \pm 0.08$ |
|                        | 10.3               | 5–20                        | 1.56                                            | $212 \pm 8$            | $0.49 \pm 0.10$ |

CNTs into the matrix area of C/C composites can induce the formation of PyC matrix with multilayer microstructures during chemical vapor deposition (CVD) [9,10], under the precondition of not increasing the F/M interface bonding. Thus, it is very possible to develop a novel method of reinforcing and meanwhile toughening PyC-based C/C composites by adding CNTs into the space between carbon fibers rather than onto fibers. In the present work, this issue was demonstrated based on the investigation into the effects of CNT growth position (on the fibers or in the spaces between them) on the flexural properties of C/C composites.

## 2. Experimental

Carbon felts (bulk density:  $0.43 \text{ g/cm}^3$ ; fibers diameter:  $7 \sim 9 \mu\text{m}$ ) were used as preform materials. The growth of CNTs in carbon felts was accomplished by catalytic CVD using  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  as catalyst precursor. Incipient wetness technique was adopted to introduce catalyst into felts using acetone as solvent. The gas system for CNT growth was  $\text{CH}_4/\text{N}_2$  (flow ratio: 1/10). The growth temperature was  $1020^\circ\text{C}$ . The growth time was 1 h. To obtain CNTs grown on carbon fibers (named CNTs-P1), before dipped in catalyst solution, carbon felts were firstly coated with a layer of PyC (several dozen nanometers) to prevent the dissolution of metal catalyst into carbon fibers and then were treated in 65 vol.%  $\text{HNO}_3$  at  $60^\circ\text{C}$  for 10 h. To obtain CNTs grown in the spaces between fibers (named CNTs-P2), carbon felts were directly dipped in catalyst solution. After CNT growth, all the felts were densified in a CVD reactor under rough laminar (RL) PyC deposition conditions [11]. For comparison, C/C composites with the same felts were prepared under the same CVD conditions. The flexural properties of the three composites were tested by three-point-bending test at room temperature, conducted at loading speed of  $0.5 \text{ mm/min}$  and support span of  $20 \text{ mm}$  length.

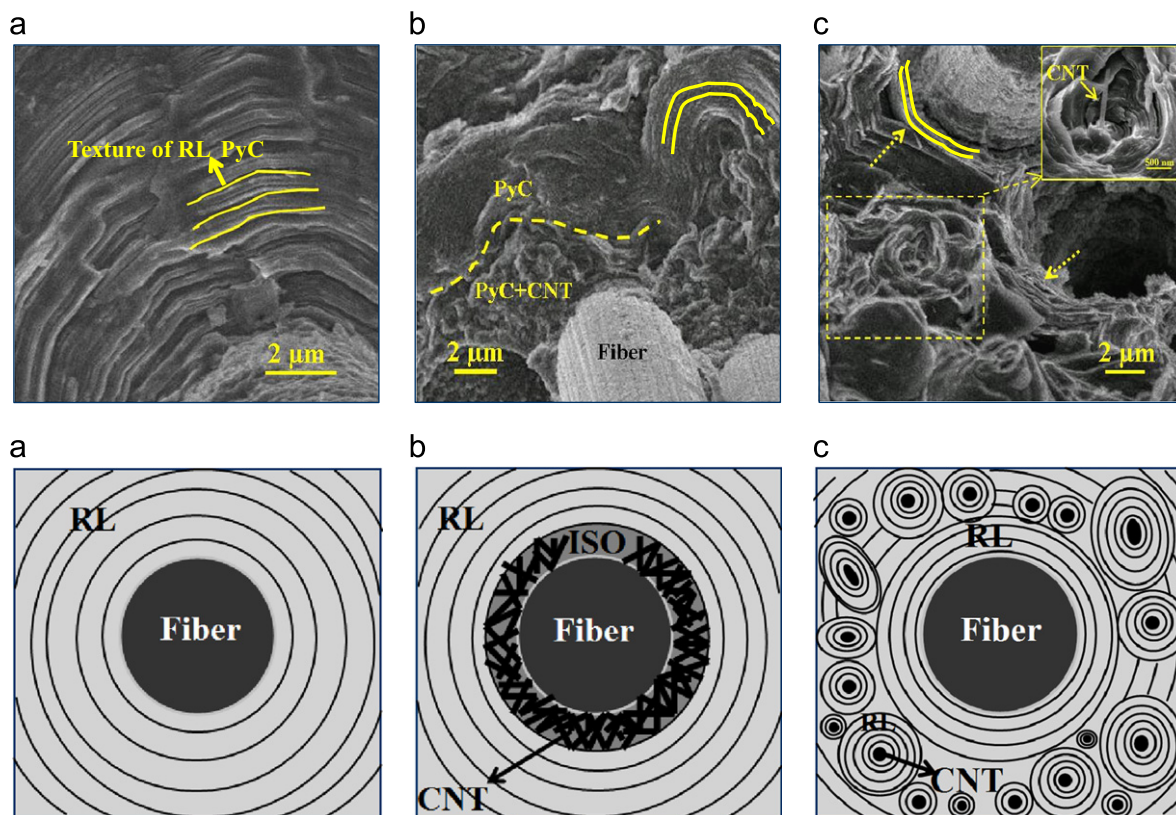


Fig. 3. SEM microstructure and its model of the three composites: (a) C/C composites; (b) CNT<sub>P1</sub>-C/C composites; (c) CNT<sub>P2</sub>-C/C composites (the inset shows that RL PyC is deposited nearly around a single CNT).

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