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The effect of SiC nanowires on the flexural properties of CVI-SiC/SiC composites

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Abstract

Single crystal SiC nanowires with volume fractions of 0%, 1.6%, 5.7% and 6.1% were incorporated in the matrices of several CVI-Tyranno-SA/SiC composites, to study the effects of the nanowires on the flexural properties of the composites. The results show that the SiC nanowires are very effective reinforcements for SiC/SiC composites. The flexural modulus, proportional limit stress and ultimate strength increase linearly with the increasing nanowires volume fraction. Specifically, the flexural properties depend on the thickness of carbon coating on the nanowires.

1. Introduction

Continuous SiC fiber reinforced SiC matrix composites (SiC/SiC) are one of the most attractive candidate structural materials for fusion because of their potential advantages for nuclear applications and superior safety characteristics compared with metallic materials [1,2]. Favorable features of SiC/SiC composites are the high temperature properties and low activation characteristics. In recent years, extensive R&D efforts on SiC/SiC have led to significant achievements in terms of reinforcement fibers, materials processing, and baseline mechanical properties and irradiation resistance [3,4]. However, the improvement of processing methods and utilization of more efficient SiC reinforcements are still necessary to obtain materials with improved thermo-mechanical characteristics and better irradiation stability. Chemical vapor infiltration (CVI) is one of the leading processes among those currently available, which allows for the production of an irradiation-resistant stoichiometric crystalline β phase SiC matrix. It is reported that [5] SiC nanowires could yield strength up to over 50 GPa. This value is far larger than those of micro-scale SiC whiskers/fibers. Therefore, SiC nanowires have great potential for use in composite materials as the reinforcements with very high strength and toughness. Recently, we have successfully incorporated single crystal SiC nanowires in CVI-SiC/SiC composite [6] and a preliminary study

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showed very effective reinforcement efficiency of the nanowires in terms of ultimate strength and toughness [7]. In this study, several CVI-Tyranno-SA/SiC composites with different amounts of SiC nanowires in the matrices have been fabricated and the flexural properties of the materials are investigated. The effects of the nanowires and their amounts on the flexural elastic modulus (E_f), proportional limit stress (σ_{PLS}), and the ultimate strength (σ_u) are specially emphasized.

2. Experimental

As shown in Table 1, four composites with different amounts of nanowires in the matrices were fabricated. Detailed information of the fabrication of SiC nanowires reinforced CVI-SiC/SiC composites has been reported elsewhere [6]. Briefly, SiC nanowires were grown in the composite preforms (multiple layers of 2D plain-woven Tyranno-SA SiC fiber clothes) prior to the CVI-SiC matrix deposition using a typical isothermal CVI system [6]. MTS (CH_3SiCl_3) was used as the source gas for SiC nanowires and matrix. MTS was carried by hydrogen. For a comparative study on the effects of the nanowires, all the composites were designed to possess 43 vol.% of Tyranno-SA fibers and 60 nm-thick pyrolytic carbon as a fiber–matrix interlayer. It is believed that similar to that of SiC fibers, when SiC nanowires are employed as reinforcement materials in SiC matrix composites, a thin compliant interfacial layer is also necessary to modify the nanowire–matrix interfacial bonding strength. Therefore, thin pyrolytic carbon layers were also deposited on the nanowires as the nanowires/matrix interphase.

Flexural properties and fracture behaviours were investigated using three-point bending test. Rectangular specimens with dimensions of $30 \times 4.0 \times 1.5\ \text{mm}^3$ were cut from the composites parallel to one of the fiber bundle directions. The bridging distance in the test was 16 mm and the loading rate was 0.5 mm/min. Due to the limitation of materials, only three bending tests were performed for each composite. Values of E_f, σ_{PLS} and σ_u were derived from the load/displacement curves according to ASTM C 1341-97 [8].

The microstructure of the nanowires and the thin carbon coating on the nanowires in the composites were examined using a field emission scanning electron microscopy (SEM, JEOL JSM-6700F) and a high resolution transmission electron microscopy (HRTEM, JEOL JEM-3000F) examinations [6,7]. The carbon interphase thickness was measured from the high magnification cross-sectional SEM images of each composite with an estimated resolution of ~10 nm. The fracture surfaces of the composites were also observed using the SEM.

3. Results and discussion

3.1. Microstructures

The density and volume fraction of nanowires of the composites are shown in Table 1. The volume fractions of the nanowires, V_{NW}, are 0 (for composite FRC, Fiber only Reinforced Composite), 1.6 (for NRC1.6, Nanowires/fibers Reinforced Composite), 5.7 (for NRC5.7), and 6.1% (for NRC6.1), respectively estimated by:

$$V_{NW} = \frac{\Delta W_p}{W_p} V_f, \tag{1}$$

where W_p is the weight of the fibers in the preform while ΔW_p is the weight gain of the preform after the nanowires process. The three nanowire composites (NRC1.6, 5.7 and 6.1) possess a similar density, ~2.6 mg/m^3, which is slightly lower than that of the conventional FRC, ~2.7 mg/m^3. The thickness of the carbon coatings on the nanowires are ~5 nm for composites NRC1.6 and 5.7, and 50 nm for NRC6.1.

Table 1
The density, volume fractions of the fiber and nanowire, carbon interphase thickness and flexural mechanical properties of the composites

Composites (ID)	Density (mg/m^3)	V_f[a] (%)	C on fiber (nm)	V_{NW} (%)	C on NW (nm)	E_f (GPa)	σ_{PLS} (MPa)	σ_u (MPa)
FRC	2.70 ± 0.14	~43	~60	0	–	140 ± 23	370 ± 77	380 ± 113[b]
NRC1.6	2.62 ± 0.03	~43	~60	1.6	~5	171 ± 35	430 ± 34	470 ± 50
NRC5.7	2.62 ± 0.03	~43	~60	5.7	~5	240 ± 35	570 ± 121	660 ± 77[b]
NRC6.1	2.61 ± 0.02	~43	~60	6.1	~50	210 ± 21	670 ± 70	750 ± 103[b]

[a] Volume fraction of Tyranno-SA fiber.

[b] Data from Ref. [7].

3.2. Fracture behaviour

Typical flexural stress–displacement curves of the composites are shown in Fig. 1, which displays several common features: (1) an initial linear region, reflecting the elastic response of the composites, followed by (2) a nonlinear domain of deformation until σ_u, likely due mainly to the matrix cracking, interfacial debonding and fiber/nanowires sliding, and individual fiber/nanowires failures, (3) quick drop(s) of the flexural stress after reached its maximum, perhaps because of the failure of a significant fraction of the fibers, and then a gradual decrease of the load. The three nanowire composites showed clear increased σ_u compared to composite FRC, in which no SiC nanowire was incorporated in the matrix.

3.3. Flexural properties

The derived average E_f, σ_{PLS} and σ_u are also listed in Table 1. Composite FRC showed average E_f, σ_{PLS} and σ_u of 140 ± 23 GPa, 370 ± 77 and 380 ± 113 MPa, respectively. All three nanowire composites showed improved flexural mechanical properties, as the amount of nanowire increased. The highest average σ_{PLS} and σ_u, 670 ± 70 and 750 ± 103 MPa, respectively, were obtained for composite NFRC6.1, which contains the highest volume fraction of nanowires, 6.1%. Composite NRC5.7 obtained the highest flexural modulus, 240 GPa, which is 100 GPa higher than that of FRC.

It is well known that the reinforcement fibers, the matrix, and the fiber/matrix interphase are several key factors determining the mechanical properties of fiber/whisker reinforced composites. As mentioned before, the amount of both the Tyranno-SA fibers and the fiber/matrix carbon interlayer were controlled to be the same in all the composites. The quality of the matrices is assumed to be similar because they were produced by the same CVI matrix densification process. Therefore, the obviously improved flexural mechanical properties of the nanowire composites are believed to be the result of the incorporated SiC nanowires. Fig. 2 graphically illustrates E_f, σ_{PLS} and σ_u versus volume fraction of nanowires. Fig. 2 shows that E_f, σ_{PLS} and σ_u increase near linearly with V_{NW} up to 5.7%, beyond which both σ_{PLS} and σ_u show a jump upwards while E_f decrease slightly. The main difference between NRC6.1 and the other two nanowire composites other than the amount of the nanowires, is the carbon coating thickness on the nanowires. The thickness of the carbon nanowire coating in NRC6.1 is ~50 nm, much thicker than that on NRC1.6 and 5.7. Therefore, the much thicker nanowire carbon coating is currently considered to be the main reason for the obvious deviation. It is well recognized that carbon coating thickness is a very important parameter affecting the mechanical properties of SiC/SiC composites. A very thin carbon coating or directly burying SiC fibers in SiC matrix composites generally produces too strong interfacial bonding to

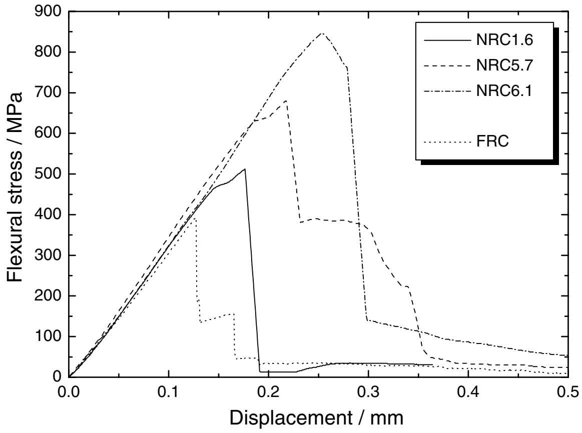

Fig. 1. Typical flexural stress–displacement curves of the composites.

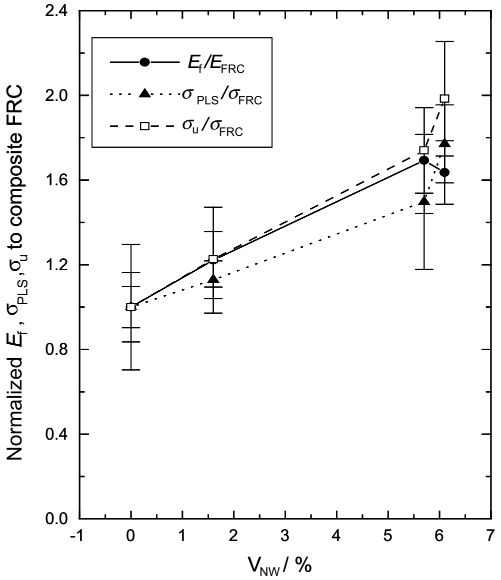

Fig. 2. Effect of SiC nanowire volume fraction on flexural modulus, proportional limit stress and ultimate flexural strength of the composites.

allow effective interfacial debonding and fiber pullout fracture behaviour, and results in unnecessarily increased strength over the bulk matrix. A similar situation is believed to occur in case of SiC nanowires. Compared with a several nanometer thick carbon coating (as in NRC1.6 and 5.7), a 50 nm carbon coating on the nanowires might produce a low enough nanowire–matrix interfacial bonding strength to allow debonding and pullout of the nanowires from the matrix to occur during failure of the composite. This hypothesis was confirmed by SEM fracture surface examinations of all the composites, as shown in Fig. 3.

Fig. 3(a) shows a typical fracture surface SEM image of NRC5.7 which reveals a sound Tyranno-SA fiber pullout fracture behaviour as in all the other composites, owing to the ~60 nm carbon fiber–matrix interlayer. However, as shown in the higher magnification image in Fig. 3(b), little debonding and pullout of nanowires is observed. In contrast, a large number of broken nanowires are readily observed at the fracture surface of NRC6.1, as shown in Fig. 3(c) and (d). Most of the debonding occurred at the carbon–nanowire interface, leaving the 50 nm carbon layer within the matrix. Such a complex fracture behaviour, with interfacial debonding, bridging/deflection of matrix cracks and deformation/pullout, and finally broking of the nanowires, is beneficial for the composite NRC6.1 to take the most advantages of the strong SiC nanowires. Therefore, the reinforcement efficiency of the nanowires in NRC6.1 is higher than those in NRC1.6 and 5.7, and results in its further improved proportional limit stress and ultimate strength. The slightly lower flexural modulus of NRC 6.1 relative to the linear relationship with volume fraction of the nanowires is likely due to the much thicker carbon nanowire coating with more compliant graphite in the matrix.

4. Conclusion

1. Silicon carbide nanowires are very effective to improve the mechanical properties of SiC/SiC composites. With ~6 vol.% of nanowires in the matrix, the composite showed near doubled ultimate strength, ~77% increased proportional limit stress, and 64% increased flexural modulus.
2. The efficiency of the SiC nanowires depends on the carbon coating thickness. Several nanometers carbon on the nanowires is not enough for the composite to take full advantages of the strong SiC nanowires. Approximately 50 nm carbon coating can produce sufficient small bonding

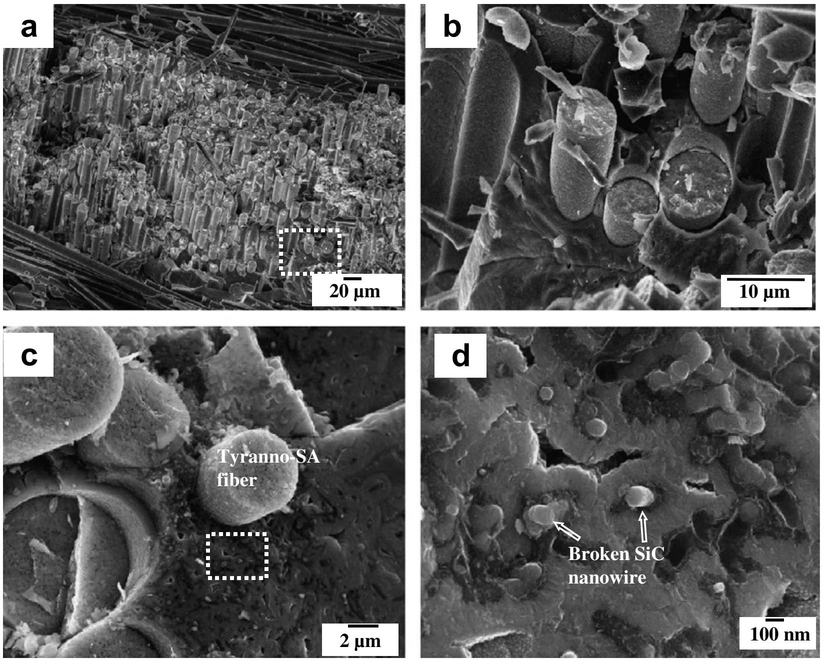

Fig. 3. Typical fracture surfaces of composite NRC5.7 ((a) and (b) showing sound fiber pullout fracture) and NRC6.1 ((c) and (d) showing debonding and pullout of the nanowires).

strength to allow moderate interfacial debonding and pullout of the nanowires from the matrix, resulting in further improving mechanical performance of the composite.

3. With the same amount of carbon coating on the nanowires, the flexural modulus, proportional limit stress and ultimate strength increase linearly with increasing volume fraction of the nanowires.

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