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Fabrication of short carbon fibre reinforced SiC multilayer composites by tape casting

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Abstract

Silicon carbide multilayer composites containing short carbon fibres (C_{sf}/SiC) were prepared by tape casting and pressureless sintering. C fibres were dispersed in solvents and then mixed with SiC slurry to make green C_{sf}/SiC tape. Triton X-100 was found to be the best one for Toho Tenax HTC124 fibres (with water soluble coating) among BYK-163, BYK-410, BYK-2150, BYK-9076, BYK-9077 and Triton X-100 dispersants. C_{sf}/SiC multilayer composites containing 5 vol.% fibre (mean fibre length of 3, 4.5, and 6 mm) were obtained. Addition of short C fibres seems to worsen the densification process in the C_{sf}/SiC multilayer composites, whereas anisotropy shrinkage in C_{sf}/SiC was also observed. Open pores size was increased slightly after the addition of C fibre but it decreased with the mean fibre length. Mechanical properties were affected by high residual porosity. The addition of short C fibre has not changed the crack deflection at weak interfaces. C_{sf}/SiC multilayer composites containing longer fibres (4.5 and 6 mm) presented higher elastic modulus, bending strength and Vickers hardness as compared to shorter fibres (3 mm). Improved sintering performance and fibre content are necessary to improve mechanical properties.

Keywords: A. Tape casting; C. Mechanical properties; Composite; Multilayer; Short fibre

1. Introduction

Silicon carbide (SiC) is one of the most promising materials for thermal protection of future reusable spacecraft, due to its low density [1], excellent high temperature mechanical properties [2] and good oxidation resistance characteristics [3]. However, similar to other monolithic ceramics, its wide application has been limited by low fracture toughness [4]. In the past, several composites (particulate- [5–7], whisker- [8], continuous [9] and short fibre reinforced composites [10]) and multilayer laminates [11,12] have been studied extensively for improving the toughness of ceramics. The key factor for toughness improvement is the presence of weak interfaces which allows energy dissipation before fracture through mechanisms of crack deflection, pull-out and bridging of fibres or whiskers, and interface delamination [3].

Continuous fibre reinforced SiC composites have received most attention in recent years. However, fabrication process of these composites is usually time-consuming and very expensive [13]. Recently, short C fibre reinforced SiC composites (C_{sf}/SiC) have been also widely studied. The use of short C fibres could reduce the cost of the composites. Moreover, the composite could be fabricated by conventional manufacturing techniques which reduce the fabrication cost. Hot-pressing [10,14–16] and sparking plasma sintering [17] have been adopted to fabricate C_{sf}/SiC composite. Multilayer structure is another effective method to improve the toughness [18]. SiC-based multilayers can display improved toughness with respect to conventional SiC providing for the required weak interfacial bonds between the layers. The weakness of interfacial bonds and residual stresses are believed to promote crack deflection phenomena, which result in the increase of the work of fracture [19].

Theoretically, coupling the above methods (multilayer with toughened single composite layer) may further improve the toughness. Zhou et al. [20] reported that the laminated ZrB_2–SiC ceramic with different SiC content layers could significantly improve the strength and toughness. Zhou et al. [20] also attributed the significant improvement in mechanical properties to the presence of residual compressive stresses arising from a mismatch in the thermal coefficient of expansion

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between the adjacent layers. Lü et al. [21] fabricated ZrB_2–SiC composites, obtained by aqueous tape casting and hot pressing, with improved mechanical properties. Lee et al. [22] reported the fabrication of oriented SiC short fibre reinforced SiC composite (SiC_{sf}/SiC) by tape casting and hot pressing. However, due to insufficient fibre pull-out, the SiC_{sf}/SiC composite showed completely brittle fracture for any fibre volume fraction [22]. Short SiC fibres were distributed rather homogeneously in the composite, which suggests that the short C fibres can also be dispersed in SiC using the similar way. In short fibre reinforced composite, short fibres could be introduced easily by conventional powder processing techniques, such as ball-milling. However, the preliminary experiment in our group showed that fibres would be cut shorter during the ball milling, which would weaken the strengthening and toughening effect of fibres [23]. Additionally, C fibre breakage would also produce large amount of residual C. Although C and B were used as sintering aids, redundant C would inhibit densification of composite [24].

Considering the protection of C fibres, mixing SiC slurry and solution containing dispersed fibres seems to be an effective method for adding fibres without breakage. The preliminary study in our group showed that uniform distribution of Toho Tenax HTA40 fibres with epoxy coating in water or organic solvent was difficult [23], and only a small fraction of fibres was dispersed, whereas the most remained aggregated in bundles at the bottom of the vial [23]. Therefore, another C fibre with more compatibility with water or organic solvent, which may show better dispersion result, was thought.

Finally, in this paper, Toho Tenax HTC124C fibres (fibre length of 3 or 6 mm) with water-soluble coating were dispersed by ultrasound in mixture of ethanol and butanol, followed by mixing with SiC slurry by mechanical stirring. Later 5 vol.% C_{sf}/SiC composites with uniform fibre distribution were fabricated by tape casting and using pressureless sintering. The effect of fibre addition on microstructure and mechanical properties was investigated.

2. Experimental

SiC multilayer and C_{sf}/SiC multilayer composite specimens were fabricated by the tape casting technique followed by pressureless sintering. The processing method involved several steps: SiC slurry preparation; fibre dispersion; adding fibre into SiC slurry; tape casting; debinding and pressureless sintering, as illustrated in Fig. 1.

The detail procedure of SiC slurry preparation has been reported in the literature [4]. In our preliminary study, Toho Tenax HTA40 fibre with epoxy coating was used as reinforcement [23,25]. However, the epoxy coating made it very difficult to disperse HTA40 fibres in a mixture of ethanol and butanol, hence a bad dispersion of fibres in SiC slurry occurred. However, in this paper, Toho Tenax HTC124 fibres (fibre length of 3 or 6 mm) with water soluble coating was used as reinforcement, which makes them easier to disperse in organic solvent. The solvent was chosen as mixture of ethanol and butanol, same as for the SiC slurry in which it has been successfully used for tape casting. The dispersion effect of BYK-163, BYK-410, BYK-2150, BYK-9076, BYK-9077 (BYK Additives & Instruments) and Triton X-100 (Sigma–Aldrich) was tested by ultrasound, as shown in Fig. 2a–f, respectively. Nonionic surfactant Triton X-100 (7 wt.%)

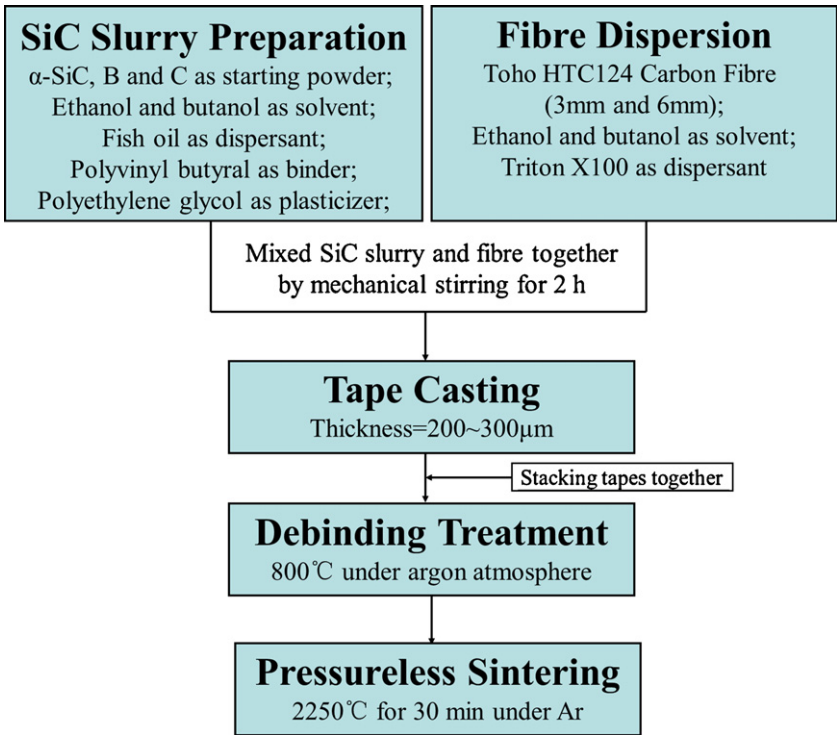

Fig. 1. Fabrication process of C_{sf}/SiC multilayer composites by tape casting and pressureless sintering.

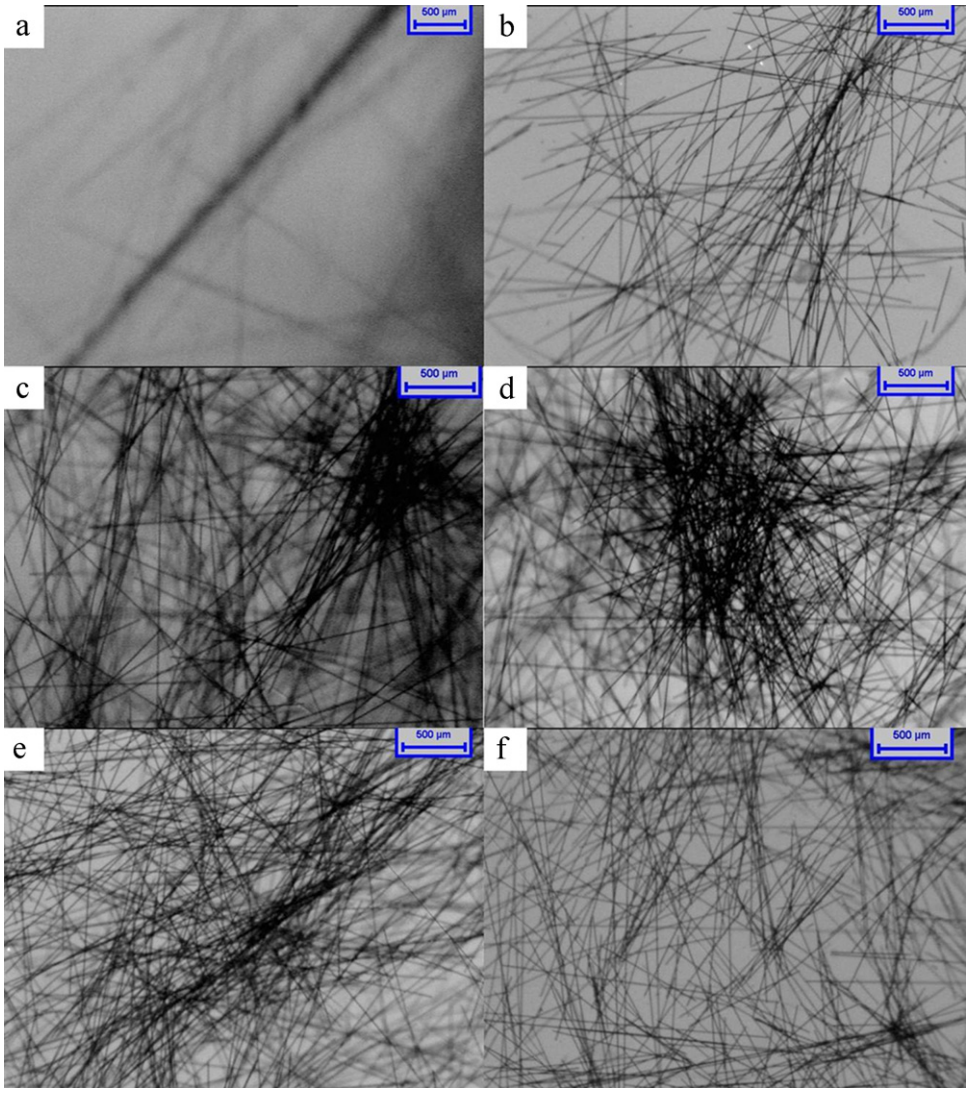

Fig. 2. Effect of dispersant (1 wt.%) on fibre dispersion (0.075 vol.%) in mixture of ethanol and butanol: (a) BYK-163, (b) BYK-410, (c) BYK-2150, (d) BYK-9077, (e) BYK-9076 and (f) Triton X-100.

showed the best dispersion result (Fig. 2f). A complete evaluation of dispersant and solvent on dispersion of fibre was out of scope of this work. It should be noted that the dispersion of fibre means to disperse C fibre separate fibre from bundles to single fibres that are still entangled. Then a good dispersion of single fibres, not entangled, can be obtained in SiC slurry only after mixing.

Afterward, the SiC slurry and dispersed fibre were mixed together for 2 h by mechanical stirring at 200 rpm in Stirrer Typer PW (Velp Scientifica s.r.l., Italy) at ambient temperature.

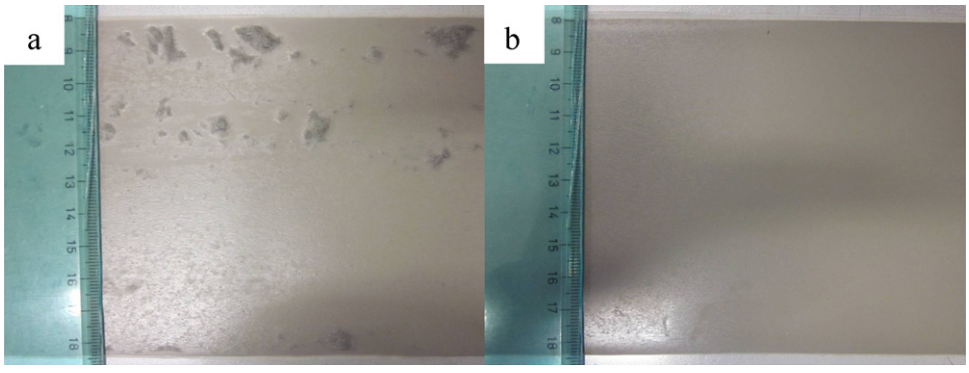

Fig. 3. Comparison of fibre dispersion in C_{sf}/SiC green tape: (a) fibre aggregation and (b) good dispersion. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1
Compositions of the SiC multilayer and C_{sf}/SiC composites.

Materials	Fibre length and content	Mean fibre length (mm)
SiC multilayer	0	–
3-C_{sf}/SiC composite	3 mm (5 vol.%)	3
4.5-C_{sf}/SiC composite	3 mm (2.5 vol.%) 6 mm (2.5 vol.%)	4.5
6-C_{sf}/SiC composite	6 mm (5 vol.%)	6

Slurry with uniform fibre dispersion was obtained. A comparison of fibre aggregation and good dispersion in green tape is shown in Fig. 3. Since the slurry containing C fibres was difficult to degas, there were many pores on the green tape, which was detrimental to the properties of final composite. Therefore, SiC slurry without degassing treatment was used to prepare SiC green tape, adopted as a reference to evaluate the effect of addition of short C fibre. The composite green tapes were carefully detached from the plastic support, cut and stacked. In order to remove the binder and the other organic components, a debinding treatment was performed up to 800 °C in argon atmosphere in Elite thermal systems limited (Tersid s.r.l., Italy). After this, the green samples were submitted to a pressureless sintering treatment in argon partial pressure (55 Pa) at 2250 °C for 30 min in vacuum furnace (TAV. S.p.A., Italy). The sintering temperature and time were based on our preliminary experiment [3]. In order to investigate the effect of fibre length on the microstructure and properties of C_{sf}/SiC composite, 3 and 6 mm short C fibres were used to obtain three composites as listed in Table 1.

Since the final materials were porous, it is difficult to measure their density by Archimedes' principle. Therefore, geometric density was used in present work. Theoretical density was calculated by the rule of mixtures. Mercury intrusion porosimetry was used for investigating the open porosity. Elastic modulus was evaluated by an impulse excitation technique involving the analysis of the transient composite natural vibration, by means of GrindoSonic MK5 instrument. The flexural strength was determined on 60 mm × 10 mm × 2 mm specimens with a span of 40 mm and a cross-head speed of 0.1 mm/min. All tests have been performed on at least five samples, in order to improve statistical significance of the results. The microstructure of samples was studied by X-ray diffraction (XRD-Rigaku D/MAX diffractometer). The microstructure and fracture surface were observed by scanning electron microscope (SEM-Zeiss Supra 25 Field Emission Scanning Electron Microscope).

3. Results and discussion

3.1. Microstructure

Cross section surface of green multilayer after stacking and fracture surface after debinding of C_{sf}/SiC multilayer composites (6 mm) is shown in Fig. 4a and b, respectively. The fibres are well distributed in the green tape (Fig. 4a and b) while no fibre bundle is observed, which also indicates the good dispersion of fibres in the tape. The pull-out of fibres is extensive due to weak bond between SiC and C fibres. Moreover, the fibres tend to align fairly well along the tape casting direction, since orientation of fibres in different directions is rarely observed. This behaviour was observed in fibre-reinforced-composite prepared by tape casting method [22,23], which implies that the properties of these composites could be designed by stacking layers with different fibre orientation.

Density and shrinkage of SiC multilayer and C_{sf}/SiC multilayer composites are listed in Table 2. Relative density of SiC multilayer in the present work (74.58%) is lower than our previous result (91%) due to the presence of pores in green tape [4], which are almost completely retained after sintering. However, it is very interesting to see that the relative densities of composite containing C fibres were higher than that of SiC multilayer, implying that the addition of C fibres seems to worsen the densification process. Fuso et al. [23] and Bolivar et al. [25] reported that the addition of carbon fibres inhibited densification process, while Lee et al. [22] reported that densification process was slightly affected by short fibres. These conflicting conclusions may indicate that the effect of fibre on densification process may be affected by fibre distribution state. Furthermore, shrinkage behavior was isotropic in SiC multilayer but not at all in C_{sf}/SiC multilayer composites. The shrinkages in length were much lower than in width and thickness in C_{sf}/SiC multilayer composites. The short C fibres tended to align along the direction of casting during the tape casting process (Fig. 4b). This result is consistent with observation of Lee et al. [22] in short SiC fibre reinforced

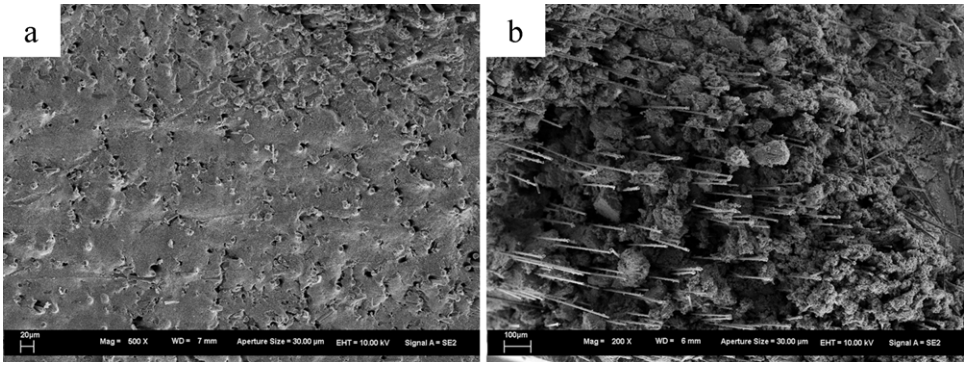

Fig. 4. Fibres were well dispersed in C_{sf}/SiC multilayer composites. (a) Cross section surface of green tape and (b) fracture surface after debinding.

Table 2
Density and shrinkage of the SiC multilayer and C_{sf}/SiC composites.

Materials	Density (g/cm^3)	Relative density (%)	Shrinkage (%)			Weight loss during sintering (%)
			Length	Width	Thickness	
SiC multilayer	2.38 ± 0.02	74.58 ± 0.5	16.6 ± 0.5	19.3 ± 0.7	18.6 ± 2.7	9.7 ± 0.3
3-C_{sf}/SiC composite	2.01 ± 0.07	64.01 ± 2.5	4.8 ± 1.9	26 ± 3.1	24.6 ± 6.4	10.8 ± 0.6
4.5-C_{sf}/SiC composite	2.24 ± 0.03	68.26 ± 0.8	6.6 ± 1.2	23.8 ± 2.7	24.5 ± 6.1	11.7 ± 0.6
6-C_{sf}/SiC composite	2.23 ± 0.07	71.18 ± 2.5	7.1 ± 1.6	24.4 ± 2.2	23.9 ± 2.8	11.3 ± 0.7

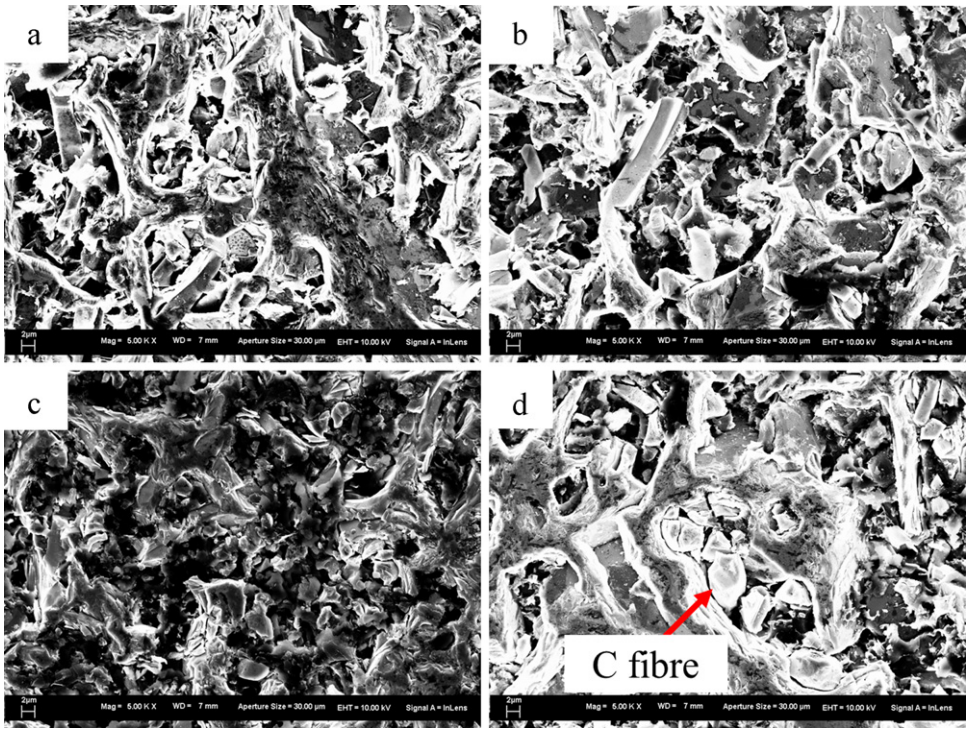

Fig. 5. Representative morphology of SiC multilayer and C_{sf}/SiC multilayer composites. (a) SiC multilayer, (b) 3-C_{sf}/SiC multilayer composite, (c) 4.5-C_{sf}/SiC multilayer composite and (d) 6-C_{sf}/SiC multilayer composite.

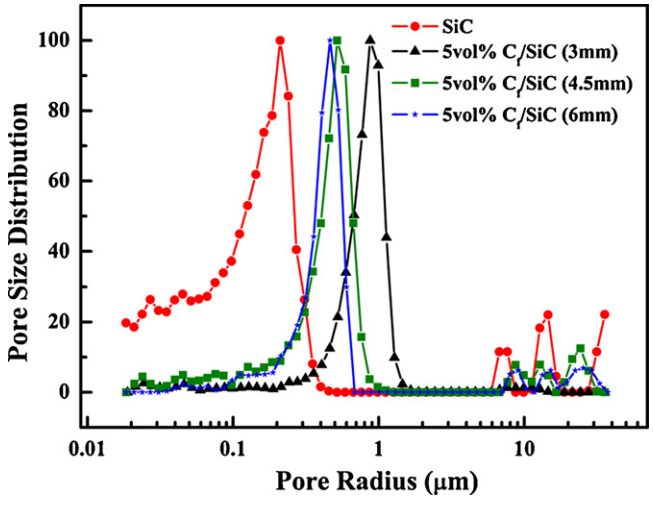

Fig. 6. Size distribution of open pores in SiC multilayer and C_{sf}/SiC multilayer composites.

composites. It implies that the shrinkage along fibre direction was restricted greatly by addition of C fibres. It is also very interesting to observe that short C fibres improve the shrinkage in width and thickness directions.

Representative morphology of SiC multilayer and C_{sf}/SiC multilayer composites are shown in Fig. 5. There are many pores in four materials which indicate the poor sintering. Moreover, few fibre was observed in composite after sintering. In order to evaluate the effect of C fibres on pore size distribution, open porosity of SiC multilayer and C_{sf}/SiC multilayer composites were tested and are shown in Fig. 6. The size of open pores increased slightly after the addition of C fibres and decreased with the mean fibre length (Fig. 6). Fig. 7 shows XRD patterns of starting SiC powder, SiC multilayer and C_{sf}/SiC multilayer composites. The peaks related to C were not clear. The C fibre content in C_{sf}/SiC multilayer composites was 5 vol.%, equivalent to 2.9 wt.%, which is lower than the limit of XRD equipment. 6H-SiC was the main phase in starting SiC powder. However, formation of 4H-SiC was detected by XRD after sintering (Fig. 7).

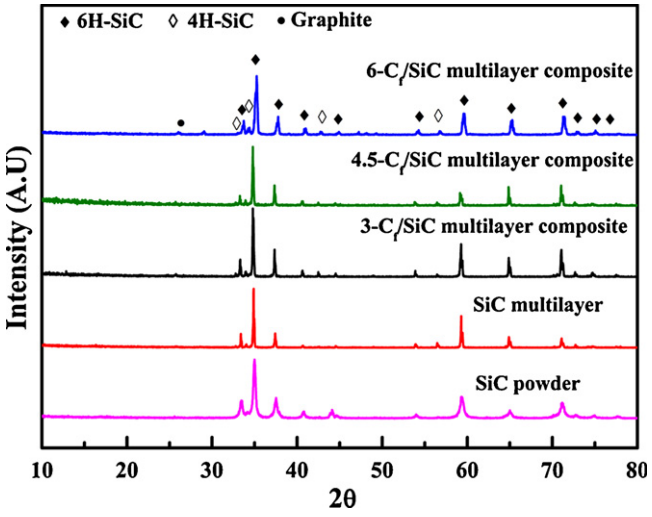

Fig. 7. XRD patterns of starting SiC powder, SiC multilayer and C_{sf}/SiC multilayer composites.

3.2. Mechanical properties

Representative bending curves of SiC multilayer and C_{sf}/SiC multilayer composites are shown in Fig. 8a. The stress/displacement curve of SiC multilayer rose up to a maximum, and then fell abruptly, corresponding to fracture of multilayer without crack deflection. This characteristic was also observed in C_{sf}/SiC multilayer composites. However, ladder-like curve, which corresponds to failure of single layer and crack deflection, can be an important characteristic for SiC-based multilayer [3,18,19]. These behaviour changes should be attributed to the poor sintering of SiC multilayer in the present study. Blanks et al. [26] calculated that the minimum volume content of porosity for crack deflection at a porous layer was 36 vol.%, which is far higher than porosity in present work. However, one sample of 3-C_{sf}/SiC multilayer composite presented ladder-like curve (Fig. 8b), and crack deflection in 6-C_{sf}/SiC multilayer composite was also observed (as shown in enlarged view in Fig. 9). These phenomena indicate that the addition of C fibres would not change the crack deflection at weak interfaces.

Table 3 summarizes elastic modulus, bending strength and Vickers hardness of SiC multilayer and C_{sf}/SiC multilayer composites. The addition of C fibre has decreased the elastic modulus as the elastic modulus of C fibre (240 GPa) is lower than that of SiC (450 GPa). The properties of 6-C_{sf}/SiC multilayer composites were close to those of SiC multilayer. A very strong relationship between mechanical properties (bending strength and fracture toughness) and relative density has been observed in SiC composite reinforced with short SiC fibre [22,27] or C fibre [14–17]. These results from literature suggest that the bending strength of these composites was limited by the amount of residual porosity. The rationale behind this hypothesis might be that the pores lower the initial microcracking stress in the matrix. Furthermore, it is obvious that C_{sf}/SiC multilayer composites containing longer fibre (4.5 and 6 mm) presented higher elastic modulus, bending strength and Vickers hardness than that containing the shorter fibre (3 mm). Sato et al. [27] observed that increasing the mean fiber

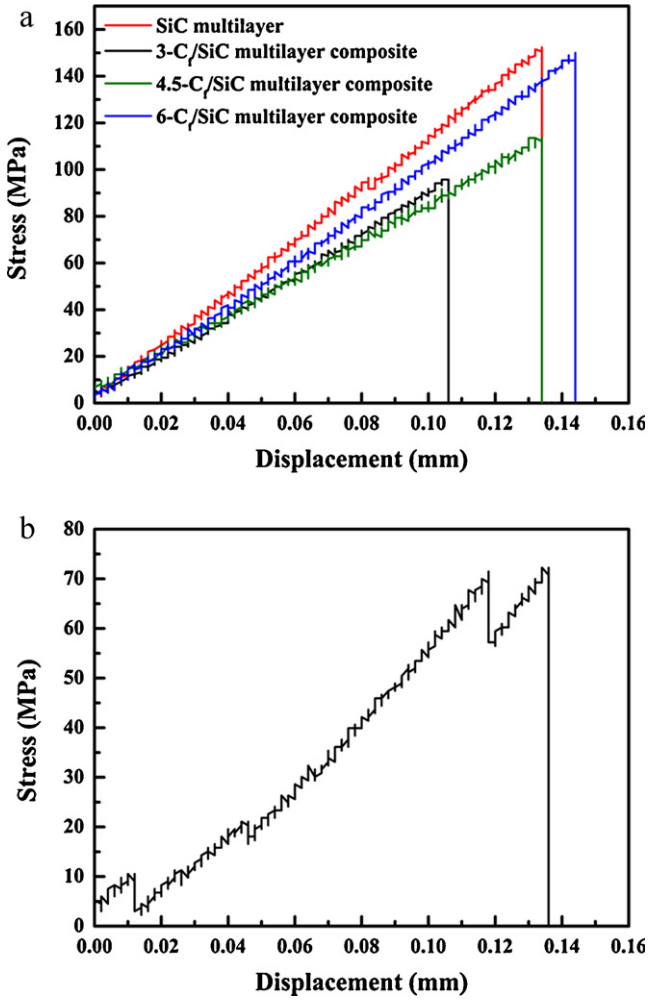

Fig. 8. Bending curves of SiC multilayer and Csf/SiC multilayer composites. (a) Representative bending curves and (b) ladder-like curve in 3-C_{sf}/SiC multilayer composite.

length from 214 to 706 μm decreased the relative density and flexural strength, which are slightly different than the present results. In the present work, the increase of mean fibre length from 3 to 6 mm has slightly increased the relative density (Table 2) and it has improved the mechanical properties (Table 3). Actually, C fibres could strengthen the composite owing to their characteristic very high strength, provided that their length is over the critical length value. These conflicting conclusions should be due to the different fibre distribution state. The fibres were distributed randomly in the work of Sato et al. [27]. However, the short C fibre tended to align along the direction of casting during the tape casting process (Fig. 4b). As reported in literature [22,23], the reinforcing effect for the random composites would be weaker than that observed for the unidirectional composites due to the presence of unemployed fibres. Anyway, improving sintering is surely needed in order to improve mechanical properties.

The fracture surface of SiC multilayer and C_{sf}/SiC multilayer composites are shown in Fig. 10. The fracture behaviour of C_{sf}/SiC multilayer composites was catastrophic where fibre pull-out was observed rarely. It indicates that the volume fraction of SiC short fibers in the present study is too low to

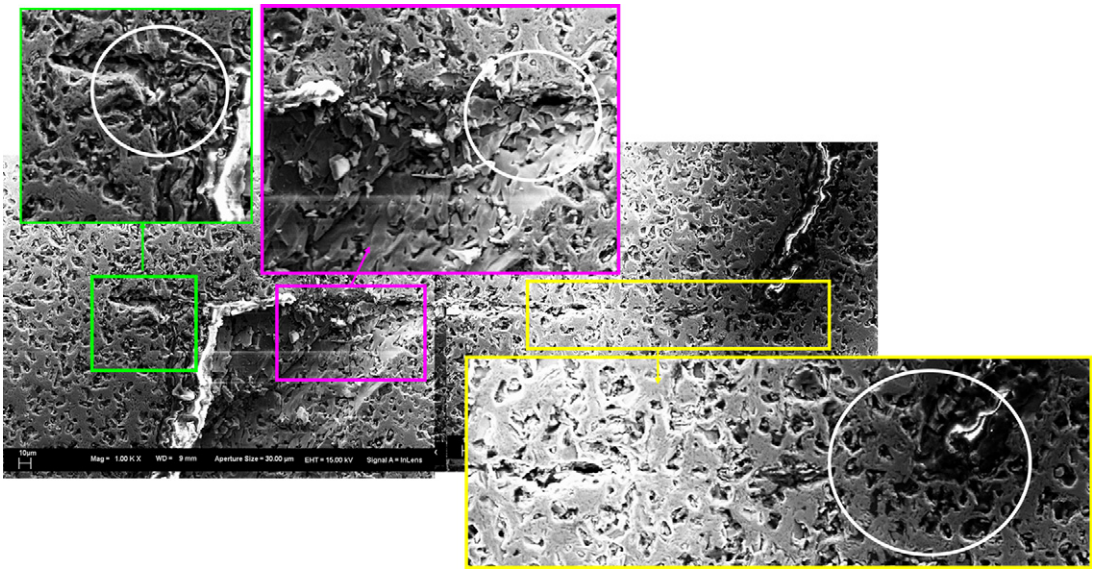

Fig. 9. Crack deflection in 6-C_{sf}/SiC multilayer composite.

Table 3
Mechanical properties of the SiC multilayer and C_{sf}/SiC multilayer composites.

Materials	Elastic modulus (GPa)	Bending strength (MPa)	Vickers hardness (HV)
SiC multilayer	197.3	154.9	856.7
3-C_{sf}/SiC multilayer composite	123.0	75.7	673.5
4.5-C_{sf}/SiC multilayer composite	163.4	104.9	766.9
6-C_{sf}/SiC multilayer composite	169.48	154.6	876.7

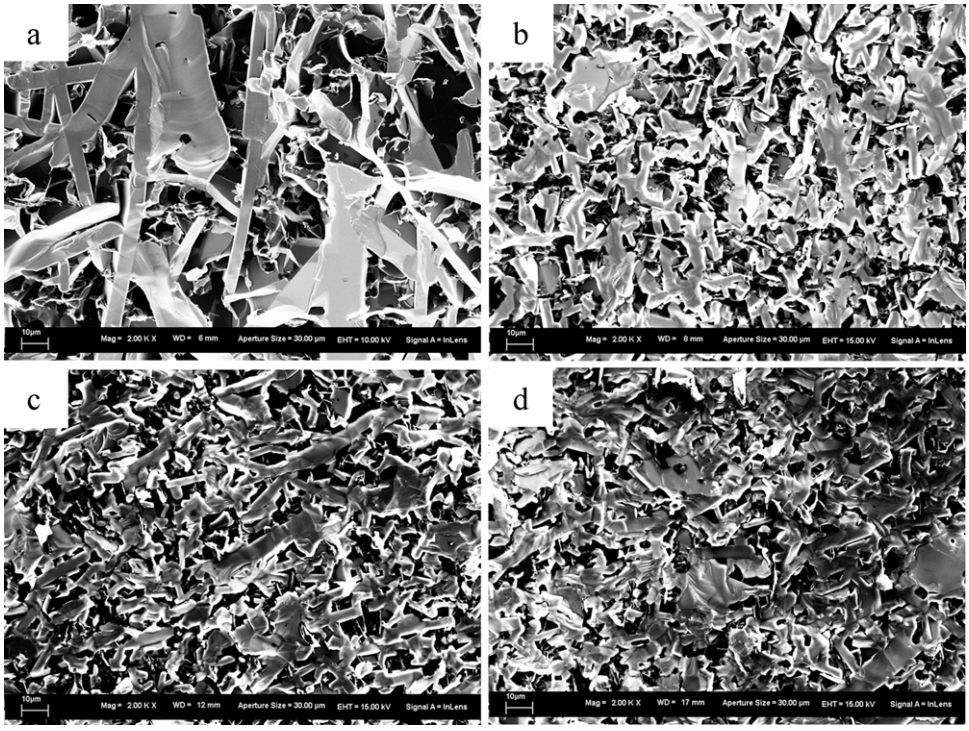

Fig. 10. Representative fracture surface of SiC multilayer and C_{sf}/SiC multilayer composites. (a) SiC multilayer, (b) 3-C_{sf}/SiC multilayer composite, (c) 4.5-C_{sf}/SiC multilayer composite and (d) 6-C_{sf}/SiC multilayer composite.

induce non-brittle fracture [27]. In case of relatively higher matrix strength, matrix cracking energy is very high. The crack approaches independent fibres, the stress cannot be sustained by fibres and results in fibre breakage without pull-out. If the fibre volume fraction in the short fibre reinforced composites is increased, matrix strength will be decreased which will change the fracture pattern of the composites depending on the interfacial bonding strength between fibres and matrix.

4. Conclusions

Silicon carbide multilayer composites containing short carbon fibres (C_{sf}/SiC) were prepared by tape casting and pressureless sintering. The C fibres were dispersed and then mixed with SiC slurry to make green C_{sf}/SiC tape. Triton X-100 was found to be the best dispersant for Toho Tenax HTC124 (with water soluble coating) among BYK-163, BYK-410, BYK-2150, BYK-9076, BYK-9077 and Triton X-100. C_{sf}/SiC multilayer composites containing 5 vol.% fibres (mean fibre length of 3, 4.5, and 6 mm) were obtained. Addition of short C fibre slightly worsen the densification process in the C_{sf}/SiC multilayer composites, whereas no isotropic shrinkage in C_{sf}/SiC multilayer composites was also observed. Open pores size was increased slightly after the addition of C fibres, but it decreased with the mean fibre length increase. Phase transformation from 6H-SiC to 4H-SiC was detected by XRD after sintering in all materials. Mechanical properties were affected by the high residual porosity. The addition of short C fibres has not changed the crack deflection at weak interface. The addition of short C fibres decreased the elastic modulus. C_{sf}/SiC multilayer composites containing longer fibres (4.5 and 6 mm) showed higher elastic modulus, bending strength and Vickers hardness than the composites containing shorter fibre (3 mm). It could be very promising to obtain C_{sf}/SiC multilayer composites with improved strength and toughness by this simple and economical method. However, the improvement of sintering process and fibre content are necessary to improve the mechanical properties.

Acknowledgments

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