Multi-wall carbon nanotubes (MWCNTs)–SiC composites by laminated technology

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Abstract

In this paper, MWCNTs–SiC composites were prepared by non aqueous tape casting and hot pressing. The dispersion of MWCNTs was investigated and correlated to the Hansen solubility parameters. The microstructure and mechanical properties of the obtained MWCNTs–SiC composites were studied. It was found that the MWCNTs were retained after sintering. An improvement in toughness was resulted with the MWCNTs content as low as 0.25 mass%. The present research provides a facile route for the preparation of ceramic–MWCNTs composites with improved properties.
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1. Introduction

Carbon nanotubes (CNTs) have attracted tremendous interest because of their superior structural, mechanical and electronic properties, such as high aspect ratio, high strength and excellent current carrying capability.[1–7] CNTs have found a wide range of applications in areas such as composite materials, scanning probe microscopy, field emission, nanoelectronics and solar cell technology.[8–10] One interesting application is to use it as the additives for making tough and stronger ceramic matrix composites.

Ma et al.[11] synthesized SiC composites containing 10 vol% of MWCNT by hot pressing a mixture of nano sized SiC and CNTs. An improvement of about 10% over monolithic SiC both in bending strength and fracture toughness was found. Similar processing was used by Siegel et al. to produce MWCT–Al$_2$O$_3$ composites.[12] Peigney and co-workers synthesized CNT–Fe/Al$_2$O$_3$ composites by growing either single wall carbon nanotube (SWCNT) or MWCNTs in situ.[13] However, the mechanical strength of the alumina composite is not enhanced, which can be attributed to the aggregation (or inhomogeneous dispersion) of CNTs in the composite materials. Chang et al.[14] fabricated the same alumina and alumina

matrix composites containing 5–20 vol% of multi-wall carbon nanotubes. An improvement of 24% on fracture toughness compared with that of the single phase alumina was found. Zhan et al. prepared nano Al$_2$O$_3$ composites containing SWNTs by spark plasma sintering (SPS), the materials showed improvement in tougheness.[15] Balázsi et al.[16] prepared Si$_3$N$_4$–CNT composites; however, the increase in strength is very limited.

A major problem in applying the nanotubes to ceramic matrix composites is how to disperse CNTs homogeneously throughout the matrix. CNTs have a strong tendency to agglomerate. Good dispersion of CNTs in a matrix of composite materials, especially in inorganic materials, indeed is a significant problem. In order to improve the homogeneity of the CNTs in the composites, Li et al.[17] prepared SWCNT reinforced SiC composites by chemical modification of MWCNT with (iodomethyl)trimethylsilane via a radical reaction and dispersed in polysilazane polymer solution. They found that high viscosity of the SWCNT/polysilazane mixtures was important for good nanotube dispersion in the Si–C–N matrix. Sun et al.[18] prepared Al$_2$O$_3$/CNTs composites by colloidal processing method, the toughness increased about 30%. Corral et al.[19] used colloidal processing and SPS sintering to prepare SWNT–Si$_3$N$_4$ nanocomposites, an increase in fracture toughness over that of the monolithic Si$_3$N$_4$ was observed for the 2.0 vol% SWCNT–Si$_3$N$_4$ nanocomposites. Ham et al.[20] found that the nanotubes were dispersed or suspended very well in the solvents with certain dispersive component (δ_d) values. Inam[21]

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used dimethylformamide as the media with ultrasonication for the dispersion of CNT and made CNT–Al_2O_3 composites, and found that the CNT was homogeneously distributed in the matrix. Other approaches to make stable dispersions include the use of surfactants,[22,23] acid treatments,[24] and chemical functionalization[25–27] that change the surface energy of CNTs, improving their adhesion/wetting characteristics and reducing their tendency to agglomerate in the solvents.[28]

Based on the theory about solubility parameters, the dispersions of nanotube in solvent can be estimated using Hansen's equation. There are three parameters in the equation:

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \tag{1}$$

where the total Hildebrand value δ_t (solubility parameter) is given by three parts: a dispersion force component δ_d, a hydrogen bonding component δ_h, and a polar component δ_p.[29] Ham et al.[20] used Hansen parameters to predict the dispersions of nanotube in solvents and found that the dispersion state partially relates to the δ_d value of the solvent. Solvent with δ_d in the range of 17–18 $MPa^{1/2}$ showed well dispersed state for CNTs.

In this paper, SiC/CNT composites were prepared by non aqueous tape casting, lamination and hot pressing. In order to disperse CNTs by colloidal processing, ethanol and methyl ethyl ketone were used as the solvent and polyvinyl pyrrolidone as the dispersant based on our previous works. After hot pressing, SiC–MWCNTs composites were obtained. The microstructure and mechanical properties were studied.

2. Experimental procedure

2.1. Processing

Commercially available MWCNTs (Shenzhen Nanotech Port Co. Ltd. (NTP), Shenzhen, China) and SiC (FCP-15, Saint-Gobain's Silicon Carbide Department, Norway) were used as the starting materials. Submicron sized alumina (Al_2O_3, Shanghai Wusong Chemical Plant, China) and yttria (Y_2O_3, Shanghai Yuelong New Materials Co., China) powder were selected as sintering additives for liquid phase sintering of SiC. MWCNTs were treated before using. Initially, MWCNTs were sonicated in 30% HNO_3 solution for 30 min, then filtered and washed with distilled water to acid-free, and finally dried at 120 °C overnight.

In consideration of the tape casting process, an azotropic mixture of ethanol and methyl ethyl ketone was selected as the solvent. The δ_d for ethanol and methyl ethyl ketone (MEK) are 15.8 and 16.0 $MPa^{1/2}$ respectively, near the defined dispersed region for MWCNTs. Therefore, the solvent mixture is expected to show well dispersed abilities for the CNTs. In the literature, ethanol has been extensively used for dispersing CNTs in different ceramic matrices.[30–32] Based on our previous study, polyvinyl pyrrolidone (PVP) was selected as the dispersant, which showed good dispersability for SiC in EtOH/MEK mixture.[33] Liu et al. showed that amides, such as *N,N*-dimethylformamide (DMF) and *N*-methylpyrrolidone (NMP), are effective for generating SWNT dispersions.[34] Therefore, PVP is also expected to be effective for CNTs, and it is more

Table 1
Slurry formulation for non-aqueous tape casting of SiC with MWCNT.

Component	Function	mass%	vol%
EtOH/MEK	Solvent	19.4–22.9	42.7–47.9
PVP (K30)	Dispersant	2.0	1.9–2.1
SiC	Ceramic powder	61.4–63.9	31.9–34.9
C nanotube	Ceramic powder	0.1–0.3	0.1–0.6
Al_2O_3	Sintering additives	1.6	0.7
Y_2O_3	Sintering additives	2. 5–2.5	0.8–0.9
PVB (B98)	Binder	4.2–4.4	6.6–7.2
Santicizer	Plasticizer	6.3–6.6	10.1–11.1

easy to obtain a co-dispersion of CNTs and SiC in the defined solvent system. For preparing tape casting slurries, a polyvinyl butyral of higher molecular weight (B98, Monsanto, USA) was selected as a binder and butyl benzyl phthalate (Santicizer 160, Solutia Inc., St. Louis, USA) as plasticizer. The formulation of the high solid loading (up to 63.9 mass%) SiC tape casting slurries is given in Table 1.

SiC tape casting slurries were prepared as follows: initially, as treated CNT powder was dispersed in the azeotropic solvent mixture in the presence of dispersant. After ultrasonic dispersion, SiC powder was added and the slurries were ball milled for 24 h, followed by adding the binder and plasticizer and ball milled for another 48 h. After milling, the homogenous slurries were transferred to a vacuum chamber where it was de-aired. Tape casting was performed on Procast Precision Tape Casting Equipment (Division of the International Inc., Ringoes, NJ) with a blade height of 200–500 mm. Drying was finished at room temperature in air.

SiC/MWCNTs composites were fabricated by cutting and stacking SiC/MWCNT green sheets. Binder removal was conducted at 800 °C for 2 h in a vacuum oven. Then the samples were sintered by hot pressing at temperature of 1850 °C and held for 0.5 h in argon atmosphere with the applied pressure of 30 MPa.

2.2. Characterizations

The density of as-sintered SiC samples was measured using Archimedes method. The XRD patterns of as-sintered SiC samples were obtained from X-ray diffraction (Rigaku D/max 2550 V X, Cu Kα) in the 2θ range of 10–70° with a scan step of 0.02°. Three-point bending strengths were measured using a universal testing machine (Instron 5565, Instron Corp., Canton, MA) at a crosshead speed of 0.1 mm/min and a span of 30 mm. The test pieces used were rectangular, 3 mm × 4 mm × 40 mm in size. Elastic modulus was calculated by bending method based on the Chinese Standard GB/T 10700-2006. Hardness was measured by Vickers' indentation with a load of 5 kg and a holding time of 10 s (Wilson–Wolpert Tukon 2100B, Instron Corp.). The fracture toughness was achieved by the single-edge notched beam test with a crosshead speed of 0.05 mm min^{-1}. At least six samples were tested for each composition for bending strength, hardness and toughness. TEM samples were prepared by cutting 3-mm-diameter disks from the samples, then thinning the disk on a grinding wheel, followed by dimpling the thinned disk

and finally ion milling it to electron transparency. TEM analysis was performed using a JEOL 2100F electron microscope with an accelerating voltage of 200 kV. The MWCNTs before and after treatment were determined using a Fourier transform infrared spectrometer (FTIR) in the 400–4000 cm^{-1} range using a KBr wafer technique; scans were run from 4000 to 400 cm^{-1}, with data spacing of 3.857 cm^{-1}. Microstructures of the samples were observed by field emission scanning electron microscopy (FESEM, JSM-6700F, JEOL, Tokyo, Japan).

3. Results and discussion

3.1. Dispersion and tape casting of CNTs–SiC composites

The dispersion of the CNT is a key issue for preparation of ceramic–CNTs composites, since the carbon nanotubes tend to form bundles or bundle aggregations. There are several ways to improve the dispersion of CNTs in the ceramic matrix. In the present work, the commercial CNTs were initially surface treated to remove catalysis particles and to generate functional groups on the surfaces of the nanotubes. The treatment procedure can be referred to the literature.[35] After treatment, the FTIR results were shown in Fig. 1.

After treatment, the absorbance bands at about 1115, 1628, 2925 and 3439 cm^{-1} were strengthened, which can be attributed to the C–O, C=C, C–H stretching and O–H stretching respectively. The enhanced peak at 1628 cm^{-1} may also come from the conjugation of C=O with C=C bonds and leads to the upshift of C=C bond.[36] The results showed the formation of carboxylic groups after treatment. These carboxylic groups are expected to
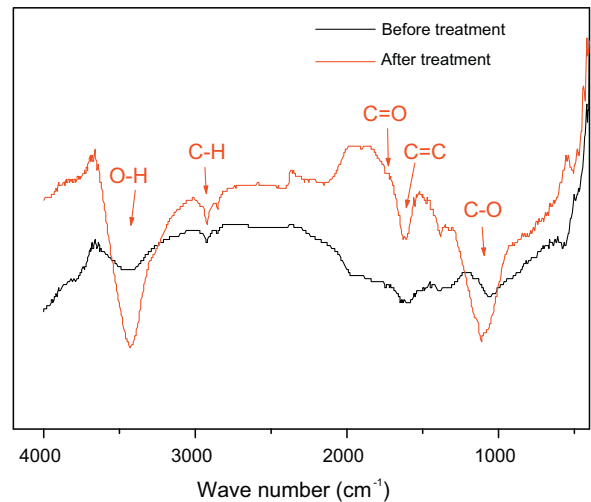

Fig. 1. FTIR spectra of MWCNTs before and after treatment.

connect with organic materials by forming covalent bondings and increase the dispersion state of nanotubes.[37] This is beneficial for the nanotube dispersions because the surface characters are changed and the van der Waals forces among nanotubes are eliminated. The formulation for tape casting of SiC/CNT was shown in Table 1 after screening.

3.2. Microstructure and mechanical properties of the composites

Fig. 2 shows the fracture surfaces of pure SiC and composites containing MWCNTs up to 0.5 mass% (Fig. 2a–d). The nano-
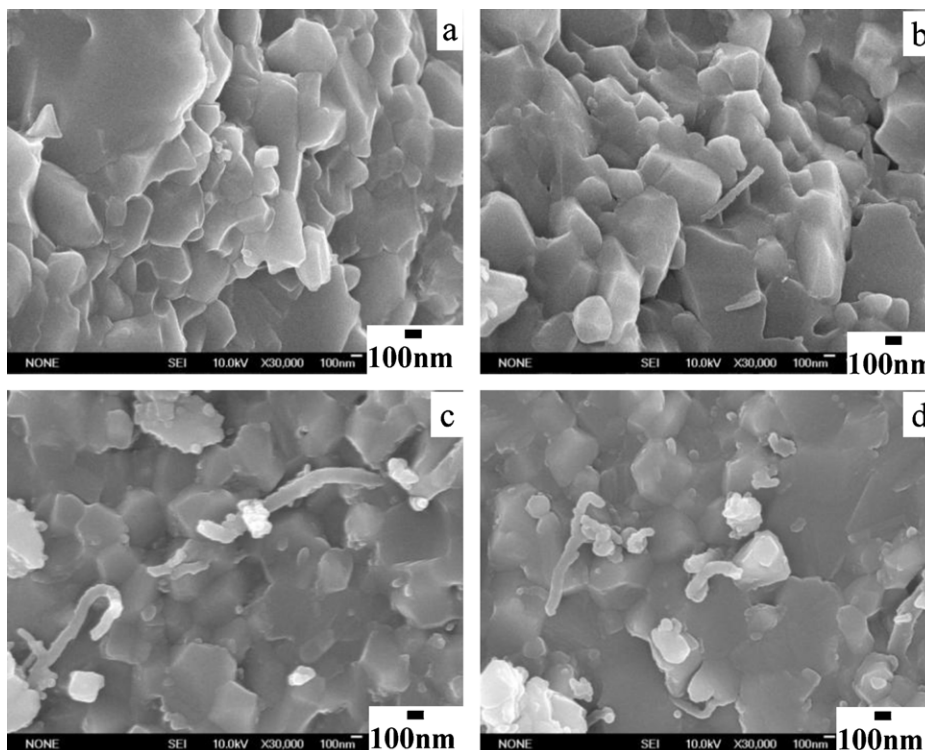

Fig. 2. Fracture surface of SiC samples with different MWCNT content: (a) no CNTs; (b) 0.1 mass% CNTs; (c) 0.25 mass% CNTs; (d) 0.5 mass% CNTs.
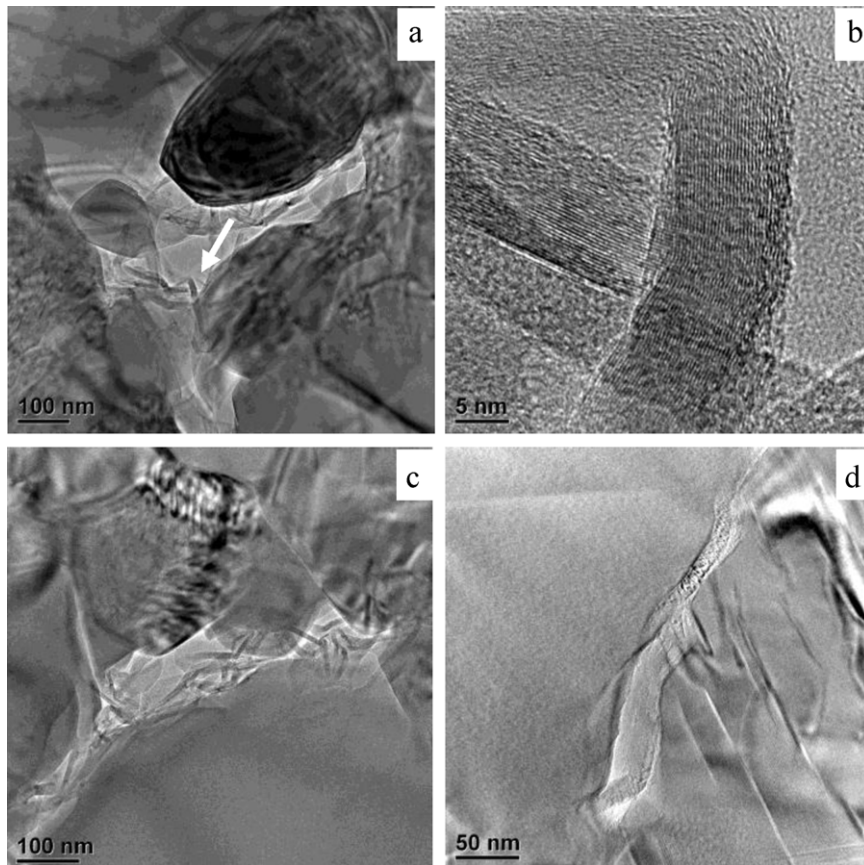
Fig. 3. TEM photographs showing the presence of MWCNT after sintering for (a and b) 0.25 mass% MWCNT–SiC; (c and d) 0.5 mass% MWCNT–SiC composites.

tubes were retained after sintering and well-dispersed at contents up to 0.5 mass%. However, the pull-outs occurring are not so frequency, it is possible that the bonding between SiC and carbon nanotube is relatively strong.

TEM images (Fig. 3a and b) of the composites revealed the presence of MWCNTs, demonstrating that they had survived the processing and heat treatment. Most of the nanotubes were found at the triple and multiple-grain junctions, at somewhat aggregation state. Fig. 3c and d shows the carbon nanotubes at the SiC grain boundary; high magnification image confirmed the strong bonding between CNT and SiC (Fig. 3d). As carbon is also the sintering additives for SiC, the strong bonding between SiC and CNT can be expected.

XRD spectra, as obtained with hot-pressed SiC–MWCNT composites, are presented in Fig. 4. The SiC substrate is 6H phase with trace $YAlO_3$ second phase. The 6H SiC represents a structure with 6-layered repeat period along the c direction and a primitive hexagonal lattice. This phase is stable and is usually observed for SiC developed at high temperature. The $YAlO_3$ phase is expected to form during the sintering process due to the loss of alumina as referred in our previous study.[38]

The relative densities of the hot-pressed SiC and SiC–MWCNT composites are shown in Table 2. The unreinforced SiC could be sintered to 97.7%, a slightly lower densities (96–97.4%) were measured for the SiC–MWCNT composites. This might suggest that MWCNTs play a negative role on the densification of SiC. In accordance with the
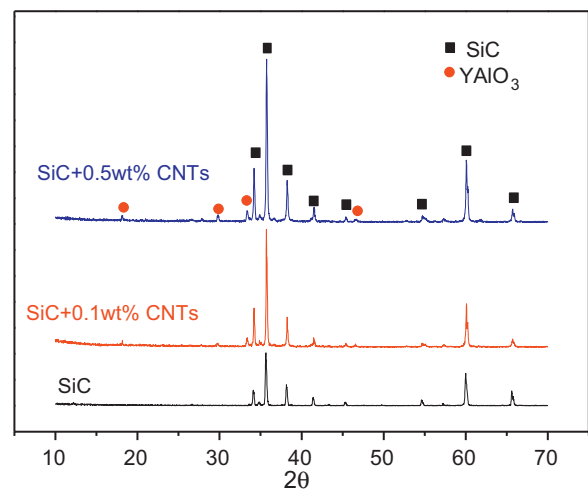

Fig. 4. XRD patterns of SiC samples with different MWCN content.

results of density measurements, the nearly absence of residual porosity can be observed for hot-pressed unreinforced SiC and SiC–MWCNT composites by scanning electron microscopy (SEM) (Fig. 2).

The hardness results obtained are reported in Table 2. The incorporation of MWCNTs in the SiC ceramics led to a modest reduction in hardness. The slightly lower sintered densities of the MWCNT-containing SiC ceramics may be partly responsible for this lowering of hardness. Such reductions in hardness on

Table 2
Mechanical properties of SiC samples with different MWCN content.

MWCT content (mass%)	Density (g/cm^3)	Strength (MPa)	Toughness (MPa m$^{1/2}$)	Elastic modulus (GPa)	Hardness (GPa)
0	97.7 ± 0.4	763 ± 79	6.7 ± 0.4	451 ± 48	25.0 ± 1.5
0.1%	96.6 ± 0.3	688 ± 58	7.3 ± 0.5	464 ± 33	24.5 ± 0.8
0.25%	96.7 ± 0.3	642 ± 91	8.7 ± 0.5	442 ± 55	24.4 ± 1.7
0.5%	97.4 ± 0.3	755 ± 106	6.1 ± 0.6	516 ± 78	23.8 ± 2.5

the incorporation of CNTs in inorganic matrices have also been observed by others.[39]

Table 2 also shows the elastic modulus (*E*) results as a function of MWCNT content. At low MWCNT content, the elastic modulus was almost kept constant though the strength decreased. At 0.5 mass% MWCNT content, however, the modulus increased, which suggests that some load transfer from the matrix to the CNTs is possible during the application of stress, though it is only a small amount.

Assuming that CNTs are distributed randomly, it is possible that at most one-third of the nanotubes are aligned along the stress direction to bear any load, while the remaining two-thirds are assumed to act as defects with negative effect to the elastic modulus. The lower values of the experimentally determined elastic modulus for the composites, as compared to even such a conservative estimate, imply that the carbon nanotubes did not effectively show the reinforcement, probably due to the agglom-

eration state of them in the SiC matrix, resulting in ineffective load transfer.

The variation of fracture toughness with MWCNT content is presented in Fig. 5a. The fracture toughness improved slightly with the incorporation of MWCNTs, reaching a maximum of 8.73 MPa m$^{1/2}$ at 0.25 mass% MWCNT content, as compared to 6.71 MPa m$^{1/2}$ for that of unreinforced SiC samples.

The flexural strengths of the unreinforced SiC ceramics and the SiC–MWCNT composites are presented in Fig. 5b. The flexural strength decreased with MWCNT content. However, at high content of 0.5 mass%, it increased to the same level as that of unreinforced SiC ceramics. This might suggest the strong bonding between MWCNT and SiC, as shown in Fig. 6. The high resolution image confirmed this. As shown in Fig. 6b, the interface between nanotube and SiC substrate is clean and the load transfer is highly possible. However, as the fracture mode for SiC–MWCNTs composites is a mixture of transgranular and
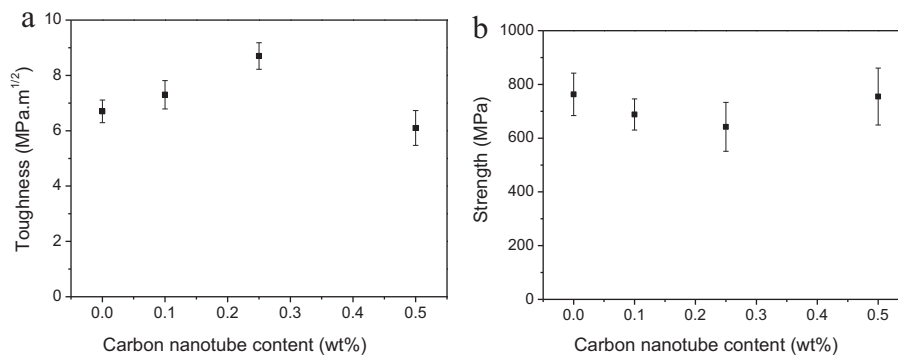

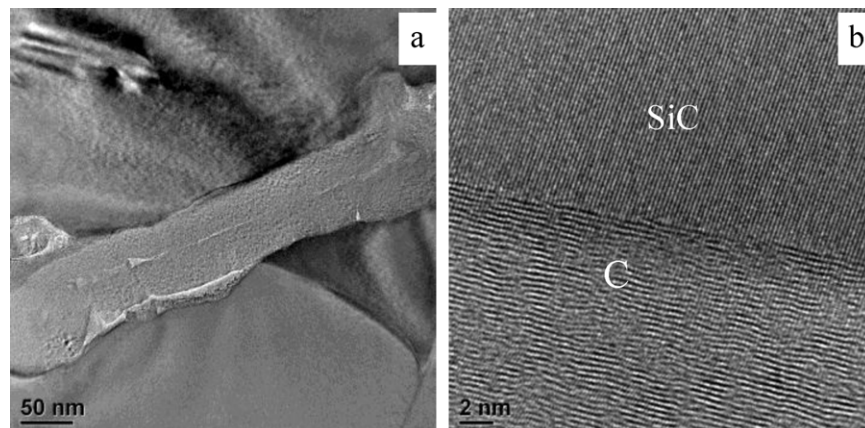Fig. 5. Mechanical properties of SiC–MWCNT composites.

Fig. 6. TEM micrographs showing the clean interface between MWCNT and SiC for SiC–0.5 mass % MWCNTs samples.

intergranular type, and the MWCNTs in SiC–MWCNTs composites are in agglomerated state, this strong bonding has very limited effects on the strength increase in SiC–MWCNTs composites.

For the SiC–0.5 mass% MWCNTs samples, the appearance of nanotubes inside SiC grains becomes significant (Fig. 2d), possibly due to the fact that the grain growth rate of SiC is fast during sintering and some nanotubes were trapped inside grains. This strong bonding between trapped nanotubes and SiC matrix will contribute to the increase in the strength. Further work on the microstructure development and the mechanical properties is in process.

4. Conclusions

The present research investigates the effects of MWCNT reinforcements on the mechanical behavior of SiC ceramics. Based on the observations and the analysis, it was found that dense CNTs/SiC composites can be developed with homogenously distributed CNTs by colloidal processing. The nanotubes retained their integrity after sintering. Reinforcement of the SiC ceramics with 0.25% MWCNT resulted in an improvement in fracture toughness. However, no obvious pullout of CNTs was observed after sintering probably due to the strong bonding between CNTs and SiC matrix. The present work suggests a facile way to prepare ceramic–CNTs composites with improved reliability.

Acknowledgements

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