Mechanical properties of carbon nanotube–alumina nanocomposites synthesized by chemical vapor deposition and spark plasma sintering

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Article info
Article history:
Received 6 March 2008
Received in revised form 9 October 2008
Accepted 13 October 2008

Keywords:
A. Nano-structures
B. Mechanical properties
D. Electron Microscopy
E. Chemical vapor deposition

Abstract
Carbon nanotubes–alumina (CNT–Al2O3) nanocomposites with variable CNT content were directly synthesized by chemical vapor deposition (CVD). The as-grown CNT–Al2O3 mixture was densified by spark plasma sintering (SPS) at 1150 and 1450 °C. Vickers hardness of 9.98 GPa and fracture toughness of 4.7 MPam1/2 were obtained for 7.39 wt.% CNT–Al2O3 nanocomposite. The addition of CNTs gives rise to 8.4% increase in hardness and 21.1% increase in toughness over that of the pure Al2O3. The optimum amount of CNTs is considered to be able to significantly enhance the mechanical property of ceramics in composites.

1. Introduction
Carbon nanotubes in the form of single-walled (SWCNTs) or multiwalled (MWCNTs) assemblies have been the focus of considerable scientific research. The unusual mechanical properties of CNTs make them an ideal class of reinforcement for composite materials [1]. CNTs have great potential for use as a toughening agent due to their very large aspect ratio (1000–10,000) [2], low density, high rigidity (Young’s modulus of the order of 1 TPa) [3,4], and high tensile strength (up to 60 GPa) [5]. In addition, the excellent electrical conductivity (10^6 S/m at 300 K for SWCNT, >10^5 S/m for MWCNT) [6,7] and thermal conductivity (6600 W/mK for an individual SWCNT and >3000 W/mK for an individual MWCNT) [8,9] make CNTs suitable candidates to synthesize nanocomposites with new functional properties. Ceramics can sustain high temperature and high hardness, but the most noted shortcoming of ceramics is the inherent brittleness, which has limited its extensive applications [10]. Thus the incorporation of CNTs with ceramics will provide an unparalleled opportunity to form a new class of CNT–ceramics materials with enhanced mechanical property.

Many attempts have been made to improve the mechanical properties of ceramics through incorporating CNTs in ceramic matrix. Siegel et al. [11] reported that the addition of 10 vol.% MWCNTs to monolithic Al2O3 could lead to 24% increase in the fracture toughness. Ma et al. [12] showed that a SiC composite containing 10 vol.% MWNTs had a 10% increase in bending strength and toughness compared with the monolithic SiC. Zhan et al. [13] obtained a fracture toughness of 9.7 MPam1/2 from 10 vol.% SWNT–Al2O3 composite, which was nearly three times higher than that of pure Al2O3. However, Laurent et al. [14] reported a contradictory result in CNT/Fe–Al2O3 composites, where the CNT reinforcement did not show any significant effect on the CNT/Fe–Al2O3 composites and the fracture toughness of CNT/Fe–Al2O3 composites was almost similar to that of carbon-free Fe–Al2O3 composites. Earlier work by Wang et al. suggested that the CNT–Al2O3 nanocomposites with 10 vol.% CNT are as brittle as bulk Al2O3, showing almost the same toughness [15]. Recently, Balani et al. synthesized CNT reinforced Al2O3 nanocomposite coating by plasma spray technique and showed an increase in the fracture toughness of 43% and elastic modulus of 200% as compared to pure Al2O3 [16]. In the present work, the synthesis and consolidation of as-grown CNT–Al2O3 composites are discussed in detail. The Vickers hardness and fracture toughness of the CNT–Al2O3 composites as a function of the CNT content and sintering temperatures are investigated.

2. Experimental method
CNT–Al2O3 mixture was synthesized by CVD. CNTs are directly grown on Al2O3 nanoparticles by using Co(NO3)2·6H2O as a catalyst precursor [18]. To upload the catalyst precursor on the Al2O3 nanoparticles, Co(NO3)2·6H2O (98%, Sigma–Aldrich) and Al2O3 powder...
(CR30 Al2O3 powder, Baikowski Inc., purity 99.99%) were mixed in ethanol, followed by sonication for 15 min. Then, the mixture was dried at 130 °C for overnight followed by grinding into a fine powder. Composites with Co/Al2O3 at ratios of 0.5–5.0 wt.% were prepared.

For a large scale synthesis of CNT, the Co/Al2O3 powder was loaded directly into a fused quartz tube, whereas for the small scale synthesis, Co/Al2O3 powder was dispersed in a Mo boat and inserted in a fused quartz tube which was thereby placed in a horizontal tube furnace. For the large amount synthesis, a fused quartz stirrer was included in the CVD system to stir the powder to achieve a uniform exposure to the reaction gases. In this work, we refer to the composite growth by using Mo boat as 'BM' mode and by stirrer as 'SM' mode. Acetylene (C2H2), hydrogen (H2), and argon (Ar) with ratio of 1:4:6 and a total flow rate of about 1100 sccm were introduced into the reaction chamber through distilled water bubbler for CNT growth. The synthesis of CNTs was carried out at 750 °C for 15 min. After the synthesis, the CNT–Al2O3 composite material was placed in 15 mm diameter graphite die and sintered under vacuum in a SPS unit [DR. SINTER spark plasma sintering system, SPS Syntex Inc.]. The following conditions were applied for the SPS treatment: pressure 100 MPa, peak temperatures 1150 or 1450 °C, heating rate 100 °C/min, hold time at peak temperature 10 min, pulse duration 12 ms, and pulse interval 2 ms. For comparison, pure Al2O3 was also sintered at the same SPS conditions. In this work, more emphasis is on the mechanical properties of nanocomposites synthesized by SM, as they depict better CNT yield, density, and mechanical properties than that synthesized by BM.

Microstructure analysis was performed by scanning electron microscopy (FESEM, JEOL, JSM-6330F) and high resolution transmission electron microscopy (TEM, JEOL 2010F). The density of the sintered samples was measured by the Archimedes method with deionized water as the immersion medium [13]. The hardness was measured by using a Vickers microhardness tester (HXD-1000TMC, Microhardness Tester, Shanghai Taiming Optical Instrument Co.) under load of 100 g with a dwell time
of 15 s. Antis equation [19] was used to calculate the fracture toughness.

3. Results and discussion

3.1. Microstructure of the CNT–Al2O3 nanocomposites

Fig. 1 shows the FESEM image of the pure alumina powder used in the present work. The average size of the alumina particles is 100–300 nm, which is consistent with the reported data by the manufacturer. Fig. 2(a)–(d) shows the FESEM images of CNT–Al2O3 nanocomposites synthesized by SM on Co/Al2O3 with Co contents of 0.5, 1.0, 2.0, and 4.0 wt.%, respectively. In general, the content of CNTs in the nanocomposites increases with an increase in the catalyst content. In the nanocomposites synthesized from 0.5 wt.% of Co/Al2O3, very few CNTs are found and the distribution of CNTs is sparse as shown in Fig. 2(a). When the Co/Al2O3 ratio increases to 1.0 and 2.0 wt.%, the uniformly distributed CNTs and small bundles are formed in the alumina matrix (Fig. 2(b) and (c)). As the Co/Al2O3 ratio reaches to 4.0 wt.%, more CNTs and tangled clusters of CNTs are observed as demonstrated in Fig. 2(d). Due to the large quantity of CNTs in the composites synthesized from high Co/Al2O3 ratio, some Al2O3 nanoparticles are wrapped around by these CNTs. The diameter of the synthesized CNTs is about 10–40 nm, and the length is about 1–7 μm. The diameter of the CNTs is found to increase with an increase in the cobalt catalyst concentration.

For comparison, FESEM images of CNT–Al2O3 composites synthesized by BM with Co/Al2O3 ratios of 1.0 and 5.0 wt.% are shown in Fig. 3(a) and (b). Nanocomposites synthesized from 1.0 wt.% Co/Al2O3 by SM (Fig. 2(b)) and by BM (Fig. 3(a)) do not show any notable differences in the uniformity and distribution of the CNTs. The weight ratios of CNTs to Al2O3 in the synthesized composites by SM are 3.19, 7.39, 8.25, and 19.10 wt.% with respect to the Co/Al2O3 ratios of 0.5, 1.0, 2.0, and 4.0 wt.%, respectively. For composites synthesized by BM, the CNT yields for Co/Al2O3 with ratios of 1.0 and 5.0 wt.% are 5.04 and 20.5 wt.%, respectively. This result also indicates that stirring the Co/Al2O3 mixture during the synthesis can promote a high yield of CNTs.

Fig. 4 shows the TEM images of CNTs grown by SM on the 0.5 and 1.0 wt.% Co/Al2O3 (Fig. 4(a) and (b)) and by BM on 1.0 and 5.0 wt.% Co/Al2O3 (Fig. 4(c) and (d)). TEM result depicts that the CNTs are multiwalled, with 10–40 graphite layers. Some CNTs have clean and curly and the Al2O3 nanoparticles still retain their original size and do not agglomerate during the CNT growth.

3.2. Effect of CNT content on the relative density

After the synthesis of the CNT–Al2O3 nanocomposite powder by CVD, SPS was employed to consolidate the nanocomposites under the conditions as described in the experimental section. The density of the sintered CNT–Al2O3 nanocomposites measured by Archimedes method is listed in Table 1. The dependence of the density on the CNT contents of the CNT–Al2O3 nanocomposites sintered at 1150 °C and 1450 °C are also plotted in Fig. 6. Fig. 6 indicates that the densities of all the CNT–Al2O3 composites sintered at different temperatures are lower than that of the pure Al2O3. The density of the CNT–Al2O3 increases with an increase in the CNT content to a maximum, then decreases for further increase in the CNT content.

The composites sintered at 1150 °C and 1450 °C have maximum densities at different CNT contents. At sintering temperature of 1150 °C, the nanocomposite with 8.25 wt.% CNT content has a maximum density of 2.62 g/cm³, while at sintering temperature of 1450 °C, the maximum density of 3.14 g/cm³ is observed for nanocomposite with 7.39 wt.% CNT content (SM). For 5.04 wt.% CNT–Al2O3 composite by BM, maximum density of 2.24 and 3.35 g/cm³ was noticed at sintering temperatures of 1150 and 1450 °C, respectively. Comparing the CNTs grown on 1.0 wt.% Co/Al2O3 in SM and BM, the density of the SM grown specimen is slightly lower than that of the BM grown sample. This is attributed to the higher yield of light-weight CNTs in the nanocomposites synthesized by SM.

From Table 1, it is also evident that the density of the CNT–Al2O3 composites with the CNT content of <8 wt.% increases significantly with the sintering temperature and the density of the CNT–Al2O3 composites with the CNT content of ≥8 wt.% is almost the same at different sintering temperatures. It can also be seen from Table 1 that the 19.1 wt.% CNT–Al2O3 composite has a minimum density among all the samples synthesized by SM. The above results indicate that the density of the CNT–Al2O3 composites does not decrease linearly with an increase in the CNT content. As known, the porosity varies inversely with the density of the composites. On the other hand, CNTs tangle and interact with each other through van der Walls force, which makes it difficult to obtain a homogeneous dispersion of CNTs in the ceramic powders. Also clustering of CNT may play a negative role same as a pore [20]. The pore density severely affects the densification.
of the nanocomposite powders. Furthermore, the density of the CNT–Al₂O₃ is determined not only by the amount of CNTs but also by the sintering temperature. The maximum density we have obtained by SM at sintering temperature of 1450 °C is 3.14 g/cm³, which is about 79.1% of the theoretical density of Al₂O₃. It is reported that CNTs in a SiO₂ matrix act as a kind of solid impurity which prevents the flow of matrix in the course of sintering, and thereby inhibit the densification of ceramic matrix [20]. A similar sintering result was reported [21] for the CNT–Al₂O₃ composites with CNT content over 10 wt.%. In the present work, it is also speculated that the CNTs in CNT–Al₂O₃ composites will hinder the SPS densification of Al₂O₃ matrix in a similar way as discussed by An et al. [21]. However, with a improved SPS treatment conditions, it is expected to obtain a better density of the composite materials.

3.3. Mechanical properties of the CNT–Al₂O₃ nanocomposites

The Vickers hardness and fracture toughness of the CNT–Al₂O₃ nanocomposites sintered at 1150 and 1450 °C are compared with those of the pure Al₂O₃ sintered at the same SPS treatment conditions, as shown in Table 1. Fig. 7 shows the Vickers hardness of the nanocomposites as a function of the CNT content. It shows that the hardness is highly dependent on both the CNT content and the sintering temperature. At sintering temperature of 1150 °C, the hardness of the composites with different CNT contents is much lower than that of the pure Al₂O₃ (8.60 GPa), while at 1450 °C, the hardness of some CNT–Al₂O₃ samples is slightly higher as compared to the pure Al₂O₃ (9.21 GPa). For example, at sintering temperature 1450 °C, the samples with CNT contents of 7.39 and 8.25 wt.% have hardness of 9.98 and 9.52 GPa, respectively, which are about 8.4 and 3.4% increase over the hardness of the pure Al₂O₃ (9.21 GPa). If the CNT content is higher than the optimum value (5–8 wt.%), the hardness of the nanocomposites becomes lower than that of the pure Al₂O₃. In Fig. 7, the microhardness increases rapidly when the CNT content increases from 3.19 to 7.39 wt.% in the nanocomposites, then it decreases gradually with CNT content from 7.39 to 19.1 wt.% (SM). A sharp decrease in the microhardness is observed for the nanocomposites with 20.5 wt.% CNT content (BM) (see Table 1). It is evident that the maximum hardness of the composites can be reached with the CNT content in the range of 5–8 wt.%. Table 1 and Fig. 7 also show that the microhardness increases dramatically with the
sintering temperature. For instance, the 7.39 wt.% CNT–Al₂O₃ composite shows the hardness of 2.29 GPa at 1150°C and 9.98 GPa at 1450°C. Apart from the CNT content and sintering temperature, the observed increase in porosity with the CNT content above the optimal value also plays an important role in the modified hardness of composites. The increase in the CNT content above the optimal value hinders the densification of the composite, and hence accounts for the decrease in the microhardness. The increase in the CNT content enhances the probability of agglomeration of the CNTs, which in turn affects the bonding of the composite, making it loose. In the course of sintering, due to the stress transfer to the CNTs, their separation from the matrix becomes much easier, which thereby results in the reduction of the mechanical properties [20]. The reduction of density in the CNT–ceramic composite has been addressed by several reports [13,22]. But its exact interaction mechanism has not been fully understood. In terms of the densification of ceramics, elimination of pore and mass transportation through bulk diffusion or surface diffusion are two basic but crucial factors which determine the ultimate density. Therefore, the effect of CNTs on the sintering property of the ceramic composites should be mainly interpreted in the view of these two points. Moreover, it is assumed that the CNTs existing in the grain boundaries act as spatial barriers to prevent closing up of grains.

As shown in Table 1, the fracture toughness of the CNT–Al₂O₃ nanocomposites sintered at 1150°C are much lower than that of pure Al₂O₃ and even it is hard to measure, due to the poor density or high porosity at this low SPS temperature. The measured maximum fracture toughness at sintering temperature of 1150°C is 1.74 MPam⁰.⁷² for 8.25 wt.% CNT–Al₂O₃ composite, which is about half of the fracture toughness of 3.50 MPam⁰.⁷² for the pure Al₂O₃ sintered at the same temperature. The fracture toughness of the CNT–Al₂O₃ nanocomposites sintered at temperature of 1450°C increases to a maximum value of 4.7 MPam⁰.⁷² with 7.39 wt.% CNT content and then decreases significantly with the further increase in the CNT percentage in the composite (Fig. 8). The

### Table 1

Mechanical properties of CNT–Al₂O₃ nanocomposites.

<table>
<thead>
<tr>
<th>Percentage of catalyst in Al₂O₃</th>
<th>CNT content (wt.%)</th>
<th>SPS temperature (°C)</th>
<th>Measured density (g/cm³)</th>
<th>Relative density (% TD)</th>
<th>Hardness (GPa)</th>
<th>Fracture toughness (MPam⁰.⁷²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>1150</td>
<td>3.56 ± 0.02</td>
<td>89.7</td>
<td>8.60 ± 0.06</td>
<td>3.50 ± 0.07</td>
</tr>
<tr>
<td>0.5 (SM)</td>
<td>3.19</td>
<td>1150</td>
<td>3.90 ± 0.07</td>
<td>98.2</td>
<td>9.21 ± 0.32</td>
<td>3.88 ± 0.20</td>
</tr>
<tr>
<td>1.0 (SM)</td>
<td>7.39</td>
<td>1150</td>
<td>2.98 ± 0.02</td>
<td>75.1</td>
<td>2.29 ± 0.72</td>
<td>3.83 ± 0.48</td>
</tr>
<tr>
<td>1.0 (SM)</td>
<td>7.39</td>
<td>1450</td>
<td>2.19 ± 0.02</td>
<td>n/a</td>
<td>2.98 ± 1.26</td>
<td>n/a</td>
</tr>
<tr>
<td>2.0 (SM)</td>
<td>8.25</td>
<td>1150</td>
<td>3.14 ± 0.02</td>
<td>79.1</td>
<td>9.98 ± 1.26</td>
<td>4.70 ± 0.74</td>
</tr>
<tr>
<td>4.0 (SM)</td>
<td>19.1</td>
<td>1150</td>
<td>2.62 ± 0.01</td>
<td>66.0</td>
<td>2.22 ± 0.45</td>
<td>1.74 ± 0.42</td>
</tr>
<tr>
<td>1.0 (BM)</td>
<td>5.04</td>
<td>1450</td>
<td>2.73 ± 0.04</td>
<td>68.8</td>
<td>9.52 ± 1.88</td>
<td>2.47 ± 0.65</td>
</tr>
<tr>
<td>5.0 (BM)</td>
<td>20.5</td>
<td>1150</td>
<td>2.31 ± 0.02</td>
<td>58.2</td>
<td>1.01 ± 0.55</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1450</td>
<td>2.37 ± 0.10</td>
<td>59.7</td>
<td>4.85 ± 1.24</td>
<td>1.32 ± 0.32</td>
</tr>
<tr>
<td>Note: TD denotes the theoretical density of Al₂O₃.</td>
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</table>
maximum fracture toughness of 4.7 MPa m^{1/2} is about 21.1% increase with respect to the fracture toughness of 3.88 MPa m^{1/2} of the pure Al_2O_3 sintered at 1450 °C. The increase in the quantity of CNTs in the composites may result in an easier transfer of the stress and thus could account for the significant increase in the fracture toughness. As the CNT content in the sample increases further, the fracture toughness decreases, indicating that the addition of CNTs above the optimal value does not account for the toughness enhancement of the Al_2O_3. Earlier report on the composites suggested that there was no noted reinforcing effect in the in situ catalytic grown and hot-pressed CNT–Al_2O_3 nanocomposites with large CNT content, and the fracture toughness of CNT–Fe–Al_2O_3 nanocomposites was similar to that of the pure Al_2O_3 [2,22]. This result is ascribed to the damage of CNTs during hot pressing, which is usually operated at the temperature as high as 1600 °C [22] and for the duration as long as 1 h [11]. In contrast to the hot pressing, the SPS sintering was performed for only 10 min in our experiments and hence is expected to minimize the damage to the CNTs. The present work suggests an increase in the density (degree of densification) at higher sintering temperature (1450 °C). The nanocomposite with higher CNT content, (19.1 wt.% for SM and 20.5 wt.% for BM) has the lowest density, hardness and toughness at any sintering temperature. This result can be related to an increase in the porosity at large CNT content, which thereby leads to the reduction in overall density and hardness. The large CNT content also affects the sintering ability, hence leading to poor densification. Sometimes, the reduction in fracture toughness with an increase in the CNT content can be attributed to the clustering of CNT bundles and also clustering of CNTs with Al_2O_3 nanoparticles at higher sintering temperature. Recent report by K. Balani et al. suggested an improvement in the fracture toughness by 11.6% with just 0.5 wt.% in situ grown CNTs in plasma-sprayed Al_2O_3 nanocomposite coating [23].

Fig. 9(a)–(d) shows the fracture surfaces of the 7.39 and 19.1 wt.% CNT–Al_2O_3 nanocomposites sintered at 1150 °C and 1450 °C, respectively. The FESEM images confirm the survival of CNTs in the nanocomposite after the quick high temperature SPS treatment. Comparing the FESEM images of the nanocomposites before (Fig. 2) and after (Fig. 9) SPS treatment, no significant change in the morphology, quantity and distribution of CNTs is observed in the consolidated nanocomposites. From the images in Fig. 9, one can find that the composites sintered at low temperature of 1150 °C (Fig. 9(a) and (c)) have high porosity while the composites sintered at high temperature of 1450 °C (images (b) and (d)) have low porosity. The intimate contact between the CNTs and the Al_2O_3 grains in the composites sintered at high temperature will contribute to the high hardness and toughness of the composites. The loose network and entangling network of CNTs are indicated in Fig. 9(b).

Fig. 10 is a high-magnification image of the fracture surface of the 7.39 wt.% CNT–Al_2O_3 composite sintered at 1450 °C. The pull-out of CNTs which were trapped in the alumina matrix grains during SPS process has been observed. The high aspect ratio and the enormous specific surface area of CNTs [24] provide a high potential for toughening through pull-outs. The interfacial friction between CNTs and Al_2O_3 matrix grains and part of the fracture energy are related to these pull-outs [24]. The pull-outs indicate the existence of weak interfacial strength between CNTs and Al_2O_3 matrix grains, which contributes to the reinforcement. The breakages and outcrops of CNTs can also be clearly seen in Fig. 9. The fracture is possibly due to the defects in CNTs. The defects...
may reduce the stiffness of CNTs, and make CNTs easily break down in these sites when they come across with the stress transferred through the Al₂O₃ matrix [13]. The fracture is mostly intergranular for the samples with different CNT contents while the fracture of the pure Al₂O₃ is essentially transgranular [25]. However, the transgranular fracture is not expected to exist in the CNT reinforced Al₂O₃ [14]. The CNT bundles could dissipate some fracture energy for the composites with micrometer sized grains, while the porosity associated with the CNT bundles may impair the reinforcement. To achieve an efficient enhancement in the mechanical property in fiber-reinforced composites, the fiber-matrix interface should be neither too weak nor too strong [14]. This kind of interface is highly desirable in the CNT–Al₂O₃ nanocomposite so that the decohesion of the CNT–matrix interfaces can allow some energy absorption under stress via CNT pull-outs [22].

The mechanical properties of the CNT–Al₂O₃ nanocomposite are affected by several factors. First, the quantity and uniformity of CNTs in the nanocomposites will greatly affect the hardness and toughness of the composites. As shown in Fig. 7 and Fig. 8, at the minimum CNT content of 3.19 wt.%, both the hardness and toughness of the composite are lower than that of the pure Al₂O₃. With an increase in the CNT content, the hardness and toughness increase to maximum values and then decrease with further increase in the CNT content. Compared with the pure Al₂O₃, 8.4% increase in Vickers hardness and a 21.1% increase in fracture toughness have been obtained from composites containing 7.39 wt.% CNTs. Indeed, small amount of CNTs is not sufficient for improving the hardness and toughness of the CNT–Al₂O₃ composite, while the excessive amount of CNTs will hinder the densification resulting in low density, hardness, and toughness. This can be explained as that the higher quantity of CNTs in ceramics seems to favor the inhibition of the matrix grain growth and hence hamper the densification. Second, the quality of CNT will significantly affect the toughness of the composites. In our CNT–Al₂O₃ nanocomposites, the CNTs are MWCNTs. In MWCNTs, the graphene layers are only linked through weak van der Waals forces [14], and the inner layers can move easily relative to the outer layers without too much

Fig. 9. Fracture surfaces of CNT–Al₂O₃ nanocomposites (SM) sintered at different temperatures. The 7.39 wt.% CNT–Al₂O₃ sintered at (a) 1150 °C and (b) 1450 °C; the 19.1 wt.% CNT–Al₂O₃ nanocomposites (SM) sintered at (c) 1150 °C and (d) 1450 °C. The scale bar for each of these images is 200 nm.

Fig. 10. Fracture surface of the 7.39 wt.% CNT–Al₂O₃ nanocomposite (SM) sintered at 1450 °C, showing the breakage and pull-out of CNTs under stress.
load. When the MWCNTs are embedded in the Al₂O₃ matrix, only their outer layers can transfer load from the matrix to the nanotubes since their inner layers cannot bond to the Al₂O₃ matrix and contribute very little to the load transfer. As compared to the SWCNTs, the MWCNTs have more defective structures, which will reduce the stiffness of CNTs in the alumina matrix. Third, the consolidation conditions of the nanocomposites are very important in improving the hardness and toughness. To enhance the mechanical properties, a dense nanocomposite with undamaged CNTs is necessary. In this work, two different sintering temperatures, 1150 °C and 1450 °C, were tried to consolidate the CNT–Al₂O₃ nanocomposites. At 1450 °C, the highest density, hardness, and toughness have been obtained. Finally, it is expected that the sintering duration is also a very important factor that affects the hardness and toughness of the composites. In the present work, SPS treatment was performed for only 10 min to consolidate CNT–Al₂O₃ powders. In future, we aim at investigating in detail the effect of the SPS conditions on the mechanical properties of CNT–Al₂O₃ nanocomposites.

4. Conclusion

CNTs have been directly grown on the Al₂O₃ nanoparticles by CVD method. The synthesized CNT–Al₂O₃ nanocomposites have been densified by SPS at 1150 °C and 1450 °C. The density of the sintered CNT–Al₂O₃ nanocomposites increases first, and then decreases with the CNT content. The maximum density of 3.14 g/cm³, hardness of 9.988 GPa, and fracture toughness of 4.7 MPam¹/² were obtained for the 7.39 wt.% CNT–Al₂O₃ composite sintered at 1450 °C. Comparing with the hardness and toughness of pure Al₂O₃, the addition of CNTs in Al₂O₃ leads up to 8.4% increase in hardness and 21.1% increase in toughness. The entangling network between CNTs and Al₂O₃ grains and the pull-outs of CNTs at fracture surface contribute to the reinforcement in the toughness. The optimal concentration of CNTs in the composite is between 5 and 8 wt.%. A higher or lower concentration of CNTs in the composite will compromise the mechanical properties of the ceramics. The sintering temperature significantly affects the mechanical properties of the CNT–Al₂O₃ composites. The composites sintered at 1450 °C has higher density, hardness, and toughness than those sintered at 1150 °C.

Acknowledgements

W.Z. Li acknowledges the support from the National Science Foundation under the grant DMR-0548061. W.Z. Li and Q.W. Wang acknowledge the support from Air Force Office of Scientific Research (AFOSR) Small Business Technology Transfer (STTR) Funding (Contract Numbers: FA9550-05-C-0126 for Phase I and FA9550-06-C-0136 for Phase II). A. Agarwal acknowledges the support from Office of Naval Research under the grant N00014-05-1-0398.

References