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# Improving Oxidation Resistance of Carbon Nanotube Nanocomposites for Aerospace Applications

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## Abstract

Carbon nanotubes (CNTs) based materials possess strong potential to substitute various functional materials developed exclusively for aerospace applications. However, because of the low oxidation temperature of CNTs (400-500 °C), using CNT based ceramic nanocomposites in high temperature applications can be problematic. Making ceramic-CNT nanocomposites by atomic layer deposition (ALD) method and field assisted sintering technology (FAST) is a good route to improve oxidative stability of CNTs. In this study, thermo-gravimetric analysis (TGA) of alumina coated CNTs (prepared by ALD) and alumina-CNT nanocomposites (prepared by FAST) were carried out. 16% improvements were observed in the oxidation resistance for alumina-CNT nanocomposites prepared by ALD and SPS techniques. Different strategies to improve oxidation resistance are discussed.

## Keywords

Carbon nanotubes, Ceramic nanocomposites, Oxidation resistance

## Disciplines

Aerospace Engineering | Engineering | Engineering Science and Materials | Nanoscience and Nanotechnology

## Comments

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# Improving Oxidation Resistance of Carbon Nanotube Nanocomposites for Aerospace Applications

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**Abstract:** Carbon nanotubes (CNTs) based materials possess strong potential to substitute various functional materials developed exclusively for aerospace applications. However, because of the low oxidation temperature of CNTs (400-500 °C), using CNT based ceramic nanocomposites in high temperature applications can be problematic. Making ceramic-CNT nanocomposites by atomic layer deposition (ALD) method and field assisted sintering technology (FAST) is a good route to improve oxidative stability of CNTs. In this study, thermo-gravimetric analysis (TGA) of alumina coated CNTs (prepared by ALD) and alumina-CNT nanocomposites (prepared by FAST) were carried out. 16% improvements were observed in the oxidation resistance for alumina-CNT nanocomposites prepared by ALD and SPS techniques. Different strategies to improve oxidation resistance are discussed.

**Key Words:** Carbon nanotubes, Ceramic nanocomposites, Oxidation resistance.

## 1. Introduction

Since the accidental re-discovery (Monthieux and Kuznetsov, 2006) of carbon nanotubes (CNTs) by Iijima (1991), it was expected that they would play an important role in the development of ceramic nanocomposites. Adding CNTs resulted significant improvements in the electrical conductivity (Thostenson et al., 2005; Dou et al., 2006), thermal conductivity (Jiang and Gao, 2008) and the fracture toughness (Zhan et al., 2003; Balazsi et al., 2005) of ceramic nanocomposites. Ceramics are very common in various high temperature aerospace applications (Barsoum et al., 1997). At high temperatures, in order to achieve maximum benefits provided by CNTs, it is crucial to retain un-attacked raw CNTs in the composites. In this way the toughening effects characteristic to micron-scale fiber composites could be explored as well. However, because of their low oxidation temperature resistance, using ceramic-CNT nanocomposites at high temperatures is an obstacle for their commercial success. Fabricating ceramic-CNT nanocomposite by atomic layer deposition (ALD) method and spark plasma sintering (SPS) provide shielding that improves oxidative stability of the encapsulated CNTs. These two strategies are investigated in this work.

ALD provides an ideal method for depositing ultrathin films on high aspect ratio surfaces as it is independent of line of sight and self-limiting (Hakim et al., 2007). Sequential surface chemical reactions deposit highly conformal films with precise control at the atomic scale (Hakim et al., 2007). The method has been shown to be a viable technique to deposit a coating on a single CNT without adversely affecting its inherent properties (Farmer and Gordon, 2006; Zhan et al., 2008).

The SPS technique is a pressure assisted fast sintering method based on high-temperature plasma momentarily generated in the gaps between powder materials by electrical discharge during on-off direct current pulsing, which causes localized high temperatures (Yamamoto et al., 2006). Other conventional methods like hot-pressing involves longer durations and high temperatures that dam-

age CNTs, leading to a decrease or total loss of reinforcing effects without producing fully dense nanocomposites (Flahaut et al., 2000; Wood, 2003; Zhan et al., 2003; Balazsi et al., 2005). SPS is always carried out in vacuum on inert gas and CNTs have been proven to be stable under temperatures as high as 2430°C in these environments (Heer and Ugarte, 1993; Ma et al., 1998). Zhang et al. fabricated bulk CNT samples by SPS and confirmed the preservation of the phase structure and the diameter of the cylindrical tubules at high temperatures of up to 2000°C (Zhang et al., 2005). During SPS, CNTs can carry current densities up to  $10^9$ – $10^{10}$  A/cm<sup>2</sup> (compared to a typical value of  $10^5$ – $10^6$  A/cm<sup>2</sup> for superconductors), and proved their stability for extended periods of time (Wei et al., 2001; Dou et al., 2006), which makes them an ideal reinforcement for ceramics matrices like boron carbide and boron nitride that are difficult to sinter in short durations.

Most of previous reports about oxidation resistance of CNTs are based on polymer based CNT composites (Kashiwagi et al., 2002; Yang et al., 2005; Costache, 2006; Bocchini et al., 2007; Kong and Zhang, 2008). Yuen et al. (2008) prepared TiO<sub>2</sub> coated CNTs - epoxy nanocomposite and reported significant improvement in the mechanical properties as compared to the uncoated CNTs - epoxy nanocomposite. For improving field emission characteristics, ceramic layers were coated on CNTs (Heo et al., 2002; Son et al., 2003; Chakrabarti et al., 2007; Pan et al., 2007), but the authors did not discuss stability against oxidation for the coated CNTs. There are many reports discussing ceramic coatings on CNTs for enhancing electronic and electrical properties (Wind et al.; 2002, Kawasaki et al.; 2008, Cao et al.; 2004, Fu et al., 2006; Bachtold et al., 2001; Javey et al., 2004). Cao et al. (2004) coated CdS and Fu et al. (2006), Bachtold et al. (2001) and Javey et al. (2004) coated alumina on CNTs for improving electronic properties only. But to the best of authors' knowledge, this is the first paper on the positive effect of ceramics on the oxidation resistance of CNTs. Wang et al. (2006) coated 10 nm of silicon layer and reported an improvement of 18.4 % in oxidation resistance. However, superior features of CNTs may not be

fully exploited with such a thick coating. Li et al. (2008) reported that the increase in Ni catalyst during CVD growth improves the oxidation temperature of Multiwall CNTs. The catalyst composition had great effect on the CNT structure and stability, which is the focus for large-scale CNT synthesis (Li et al., 2008). CNTs were distinctly observed by Laha et al. (2004) after plasma spraying blended powder (Al-Si-CNTs) on a rotating metallic mandrel. In the same report, CNTs were sprayed at very high temperatures (9700°C – 14700°C), but for very short durations. This conference paper reports the high temperature shielding effect of alumina on CNTs in the coated and the nanocomposite systems, fabricated by ALD and SPS respectively. The oxidation resistance of CNTs in different systems was characterized thermogravimetric analysis (TGA).

## 2. Experimental procedure

### 2.1 Material

DMF was supplied by Sigma-Aldrich, UK. The CNTs used in this study were commercially available as “Multiwall carbon nanotubes (MWNTs), NC-7000” from Nanocyl Inc., Belgium. They were synthesized by the catalytic CVD method and have an entangled cotton-like form. The CNTs had an average outer diameter of 9.5 nm (10 graphitic shells), length of up to ~1.5 microns and density of 1.66 g/cm<sup>3</sup>. The alumina matrix used in this study was commercially available “544833 aluminum oxide” nanopowder from Sigma-Aldrich, UK. As supplied by the supplier, the main features of this product are: gamma phase; particle size < 50 nm; surface area 35-43 m<sup>2</sup>/g; melting point 2040°C; and density 3.97 g/cm<sup>3</sup>. Another batch of multiwall CNTs was supplied by Nanodynamics Inc., USA (figure 1a). These CNTs had an average outer diameter of 15 nm and, length of up to ~2 microns and specific surface area of 182 m<sup>2</sup>/g. Alumina coating on these CNTs by Nanodynamics Inc. was done by elsewhere by atomic layer deposition method (Hakim et al., 2007). Two coating cycles were followed. 27 cycles of ALD resulted in 25 nm of coating, whereas 54 cycles resulted in 50 nm (Fig. 1b).

### 2.2 Composite powder preparation and Spark Plasma Sintering (SPS)

Alumina + 11.2 vol% (5 mass%) MWNTs composites were prepared. Details of the processing can be found elsewhere (Inam et al., 2008). Briefly, MWNTs were dispersed in DMF via high power bath-sonication for 2 hours and then hand-mixed with alumina nanopowder for another 5 minutes. The liquid mixture was transferred to another jar filled with zirconia balls (milling media) of two different sizes (10 and 5 mm, mass ratio: 3:2). The jar was sealed and rotation ball milled for 8 hours at ~200 RPM. The milled slurry mixture was dried at 75°C for 12 hours on a heating plate and then transferred to a vacuum oven (100°C) for 3 days for complete removal of dispersant. A solvent-trap (filled with ice) was connected between the vacuum pump and the oven. The dried mixture was ground and sieved using a 250 mesh and then returned to the vacuum oven for another 4 days at the same

temperature for thorough extraction of the solvent. This lengthy drying procedure was followed because any residual solvent has a detrimental effect on the properties of CNTs reinforced nanocomposites (Lau et al., 2005; Moniruzzaman et al., 2006). Dried composite powder (~ 2 grams) was poured into a carbon die and cold pressed at 0.62 MPa for 5 seconds before sintering. Nanocomposite disks (thickness 2 mm and diameter 20 mm) were prepared by Spark Plasma Sintering (SPS) in a SPS 2040 furnace (Sumitomo Coal Mining Co, Japan). A pressure of 100 MPa was applied concurrently with the heating (rate 300°C/min) and released at the end of the sintering time, which was 3 minutes for all of the samples. The furnace has a pyrometer focused on a hole close to the sample in the upper punch to measure the processing temperature. Details of the SPS technique are reported elsewhere (Omori, 2000). The sintering temperatures were 1200°C and 1800°C. All of the samples were slowly cooled to avoid fracture due to thermal shocks and differential contractions. The densities for both sintered samples were 100% of the theoretical density.

### 2.3 Characterisations

Thermogravimetric analyses (TGA) were performed on a TA Instruments SDT Q600 TGA thermogravimetric analyzer. Samples were analyzed in platinum pans at a heating rate of 30°C/min to 1000°C in air flowing at 180 ml/min. Powder sample masses ranged from 30 to 40 mg, whereas sintered sample masses ranged from 30-50 mg. SPSed samples were fractured in order to observe the

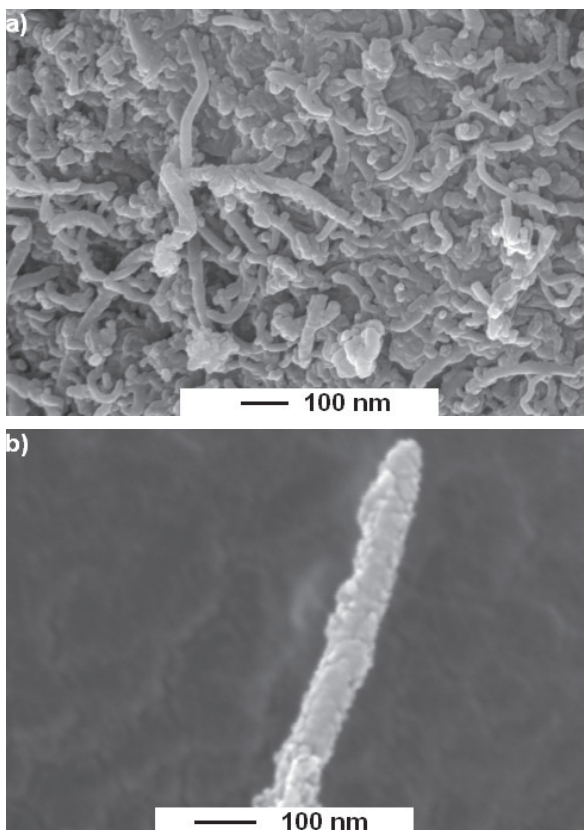


Figure 1. Multiwall CNTs: a) uncoated agglomerates; and b) single coated (54 ALD cycles) CNT.

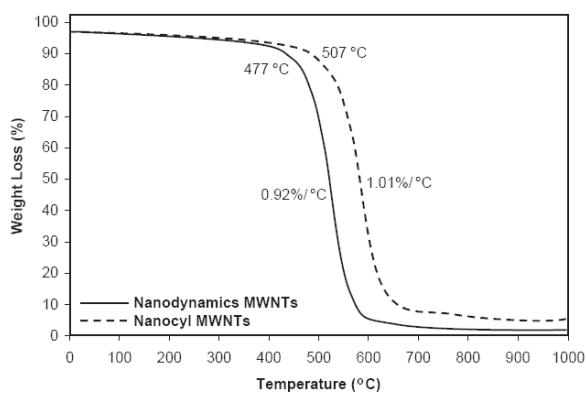


Figure 2. TGA of raw CNTs

agglomeration and dispersion of CNTs. Fractured surfaces and powder samples were gold coated and observed in a FE-SEM (FEI, Inspect F, 20 kV, working distance 8-10 mm).

### 3. Results and discussion

Fig. 2 shows the oxidation behaviour of CNTs obtained from different suppliers. CNTs provided by Nanodynamics Inc, have larger average diameter as compared to the CNTs provided by the other source. The oxidative stability of CNTs is also influenced by defects (Li et al., 2008) and nanotube diameter (Yao et al., 1998; Li et al., 2008). Oxygen molecules react easily with larger surface areas, resulting in decreased oxidative stability of Nanodynamics CNTs. The oxidation of CNTs is not rapid and acute like combustion, which is also evident in other report (Tian et al., 2007). Because the kinetic energy of oxygen varies with temperature, there is not a critical temperature when the oxidation of CNTs starts (Tian et al., 2007) as

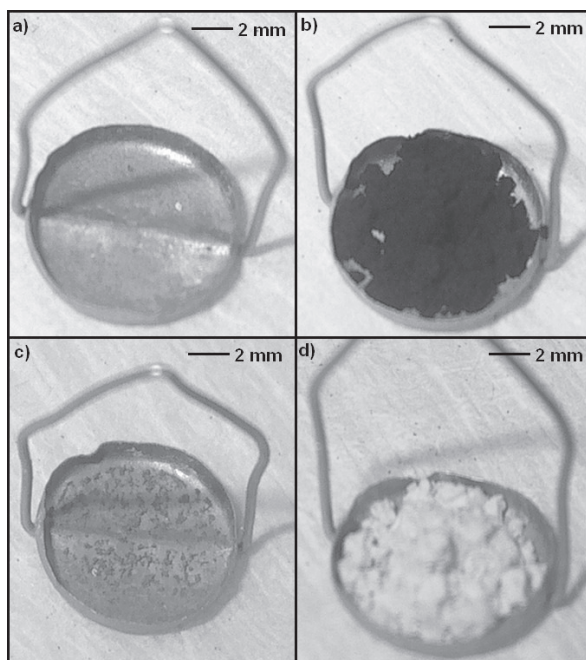


Figure 3. Platinum pan used for TGA: a) empty pan; b) CNTs before oxidation; c) impurities left after oxidation of uncoated CNTs; and d) alumina nanotubes left after oxidation of coated CNTs.

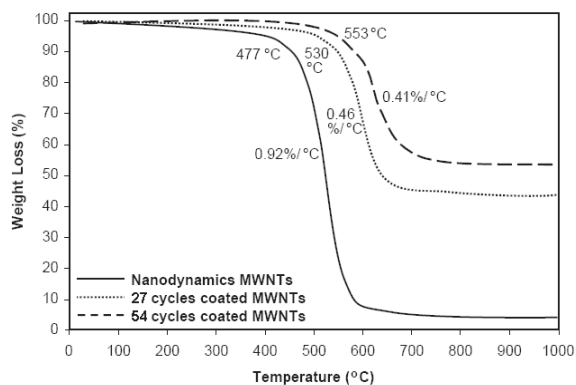


Figure 4. TGA of uncoated CNTs and alumina coated CNTs.

shown in Fig. 2. During the initial stage of TGA, all samples showed a slight mass loss due to the presence of amorphous carbon, as reported in other papers (Chen et al., 1998; Li et al., 2008). In the second stage of TGA, the curve slope is maintained almost the same in the definite temperature range for both types of CNTs. In the third stage of TGA, there was no weight gain observed during thermal treatment, since no oxidation of the impurities occurred. The weight loss for both types of CNTs was not 100 % due to the presence of impurities, which were found after the furnace pan (Fig. 3a) was cooled down (Fig. 3b and 3c).

To improve the oxidation resistance and chemical stability of CNTs, a protective film or coating was necessary to shield the CNTs against thermal or environment damage. The oxidative stability of CNTs was distinctly improved due to the protective alumina coating (Fig. 4). The onset oxidation temperature for sample coated for 54 ALD cycles is now as high as 553°C in air atmosphere, which is 76°C (16%) higher than that of uncoated CNTs. Once the oxidation started, the degradation rate was also reduced to 0.41%/°C, which is 55% less than that of uncoated CNTs. The degradation process was delayed because it became more difficult for oxygen molecule to approach CNTs after coating. By analysing the third stage (after 700°C) of the TGA (Fig. 4), it is possible to quantify the mass content of CNTs in the coated nanocomposite. 27 ALD cycles resulted in 56.4 mass% of CNTs and 54 ALD cycles produced 44.9 mass% of CNTs in the coated nanocomposites. A thicker alumina coating could further inhibit the oxidation of CNTs but it may decrease the mechanical properties of CNTs by making the coated CNTs brittle. After cooling down the TGA furnace, white coloured alumina nanotubes (Fig. 3d) were left in the platinum pan, which were previously surrounding CNTs. This could be one of the ways to mass-produce these alumina nanotubes (Fig. 5).

Dense CNT-dispersed alumina nanocomposites with different grain sizes were fabricated using SPS. In figure 6, the onset oxidation temperature for sample coated for 54 ALD cycles is now as high as 588°C in air atmosphere, which is 81°C (16%) higher than that of raw CNTs. Once the oxidation started, the degradation rate was also reduced to 0.026%/°C, which is 97% less than that of raw CNTs. Representative images of the fractured surfaces of the SPSed nanocomposites were selected for studying the grain sizes (Fig. 7). Oxidative reactivity in

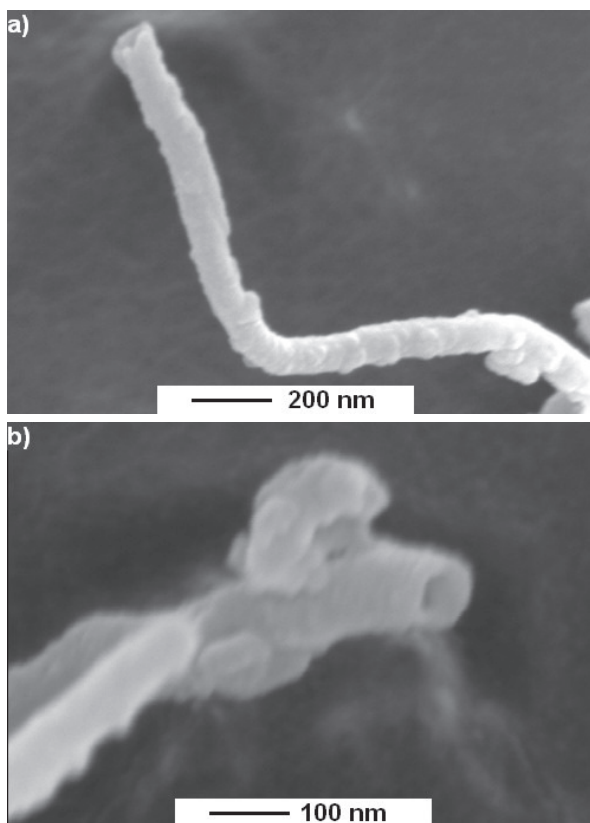


Figure 5. Alumina nanotube left after the oxidation of coated CNTs: a) at lower magnification; and b) at higher magnification.

these nanocomposites is dominated by the alumina grain size. Sample sintered at 1200°C produced finer grains (Fig. 7a). This produced large area of grain boundaries or easy entry path for oxidation reaction. However, in coarse-grained nanocomposite sintered at 1800°C (Fig. 7b), fewer grain boundaries made difficult for oxygen to approach CNTs, leading to a better oxidative stability.

#### 4. Conclusion

It is necessary to preserve the chicken wire hexagonal structure of CNTs in ceramics for high-temperature applications. Fabricating ceramic-CNT nanocomposite by ALD method and SPS provide shielding that improves oxidative stability of the encapsulated CNTs. In this study, 16% improvements in the oxidation resistance were

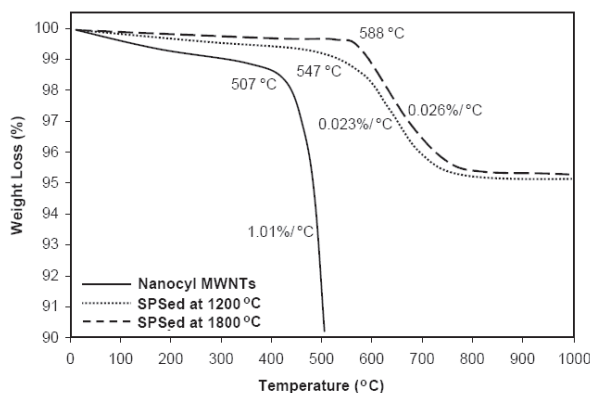


Figure 6. TGA of SPSed nanocomposites and raw CNTs.

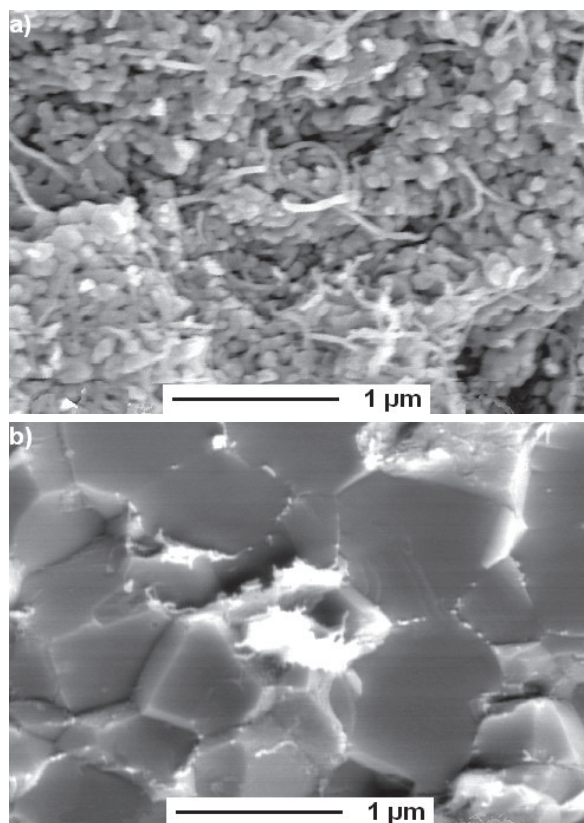


Figure 7. Representative fractured surfaces of the SPSed samples: a) SPSed at 1200°C; and b) SPSed at 1800°C. Bright area represents CNTs.

observed for alumina-CNT nanocomposites that could be further improved by varying processing conditions. In ALD, a thicker alumina coating could further inhibit the oxidation of CNTs and enhance the thermal stability of CNTs. Oxidising CNTs after coating them with ceramics could be one of the ways for mass-production of ceramic nanotubes. In SPS, coarser grains protect CNTs more efficiently as compared to the finer ones, due to the presence of fewer grain boundaries. It was found that TGA is a good tool to evaluate the mass content of CNTs in the coated CNT and SPSed nanocomposites.

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