

Characterization of carbon nanotubes and carbon nanotube-epoxy composites

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Abstract

The mechanical and electrical properties of carbon nanotube composites are attractive for a number of applications. The final properties depend on the quality of the feed stock, the amount of material used, and methods for de-aggregating and dispersing the nanotubes. Here, Fourier transform infrared spectroscopy, RAMAN spectroscopy, scanning electron microscopy, thermogravimetric analysis, and permittivity measurements are used to discern between three commercially available multi-walled carbon nanotubes (Nanocyl 7000, Cheap Tubes, and a sample of acid functionalized multi-walled nanotubes), and their epoxy composites. The RAMAN and thermogravimetric analysis data indicated that the Nanocyl had the most defects and impurities, however, the permittivity measurements and scanning electron microscopy data indicated that the Nanocyl nanotubes were well distributed in the matrix. The most important factors for dispersion and high permittivity appear to be the ability to de-aggregate the nanotubes, which is enabled by the short length, and a small diameter. The small diameter results in an overall longer length of carbon nanotube in the composite compared to an equal mass of larger diameter nanotubes.

Significance to defence and security

Carbon nanotubes can improve the mechanical and electrical properties of composite materials, impacting their strength and flexibility, and permitting the development of sensors and actuators, with low filler loading. The development of microwave absorbers for signature management is an example of a military application for these materials. The permittivity values can be used for designing absorbing materials, however, the designs are sensitive to the material properties which are affected by the quality of the material supplied, and by processing. The methods described here can be used to reduce these effects.

Résumé

Les propriétés mécaniques et électriques des matériaux composites à base de nanotubes de carbone sont intéressantes pour un certain nombre d'applications. Les propriétés finales de ces matériaux dépendent de la qualité des matières premières, de la quantité utilisée et des méthodes de désagrégation et de dispersion des nanotubes. Pour le présent travail, nous avons utilisé la spectroscopie infrarouge à transformée de Fourier, la spectroscopie RAMAN, la microscopie électronique à balayage, l'analyse thermogravimétrique et des mesures de permittivité pour faire la distinction entre trois nanotubes de carbone multiparois disponibles sur le marché, (Nanocyl 7000, Cheap Tubes et un échantillon de nanotubes de carbone multiparois à fonctionnalité acide) et leurs composites époxydiques. Les données des analyses RAMAN et thermogravimétrique ont montré que le Nanocyl comportait le plus grand nombre de défauts et d'impuretés. Cependant, les mesures de permittivité et les données de la microscopie électronique à balayage ont indiqué que les nanotubes Nanocyl étaient bien répartis dans la matrice. Les facteurs de dispersion et de permittivité élevée les plus importants semblent être la capacité de désagrégation des nanotubes, qui est activée par une courte longueur et un petit diamètre. Le petit diamètre entraîne une plus grande longueur globale des nanotubes dans le composite comparativement à une masse égale de nanotubes de plus grand diamètre.

Importance pour la défense et la sécurité

Les nanotubes de carbone peuvent améliorer les propriétés mécaniques et électriques des matériaux composites, parce qu'ils influent sur leur résistance et leur flexibilité, et permettent la mise au point de capteurs et d'actionneurs ayant des chargements de charges moindres. La mise au point d'absorbants de micro-ondes à des fins de gestion de la signature est un exemple d'application militaire de ces matériaux. Les valeurs de permittivité peuvent être utilisées pour concevoir des matériaux absorbants. Néanmoins, les conceptions sont sensibles aux propriétés de matériaux, qui sont affectées par la qualité des matières fournies et par le traitement. Les méthodes décrites dans le présent travail peuvent être utilisées pour réduire ces effets.

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1 Introduction

It has been demonstrated that the addition of high length-to-diameter aspect ratio carbon nanotubes to polymers improves a composite material's mechanical [1], electrical [2], and thermal [3] conductivity properties. Potential applications for these composites include microwave absorbers [4], sensors [5], actuators [5], and supercapacitors [6]. A good deal of research has been conducted into developing the capability to reproducibly achieve consistent composite properties, which depend on the quality of the nanotube feed stock, the amount of the material used and the methods used to separate and disperse the nanotubes [7]. The mechanical methods used to separate carbon nanotube agglomerates tend to fracture the nanotubes into shorter pieces. In addition, methods used to disperse the nanotubes into the polymer involve shear which can further fracture the nanotubes, and shear rates have been found to affect the nanotube dispersion [7]. Both of these factors have significant implications to the composite's properties. For example, a percolating conductive network could be formed for a composite as a result of a uniform distribution of high aspect ratio carbon nanotubes. If the processing decreases the length of the nanotubes, even though they are well distributed, not enough nanotube-nanotube contacts may occur to give a conductive network until higher loadings are reached. On the other, hand shear effects, occurring during the dispersion of the nanotubes, can result in the formation of high and low nanotube density regions, where the nanotubes in the high density region form a conductive network so conductive networks are reached at lower loadings.

The purity, functional groups and defects in the nanotubes also impact the composite's properties. Carbon nanotubes are based on graphitic sheets that are rolled up into tubes. Single layer tubes are referred to as single-walled nanotubes (SWNTs), and multiple layers as multi-walled nanotubes (MWNTs). The unique conductivity and strength properties of these materials are a consequence of the graphitic structure, and these are degraded if defects are present such as broken sp^2 bonds or the presence of sp^3 -bonding [8]. In addition nanotube fabrication methods can result in the product containing catalyst, and amorphous and mesographitic carbon. The amount of defects and impurities in a sample affect the composite's properties as do the nanotube diameter and length, and how well the tubes are dispersed in the resin as discussed above.

Conductivity and permittivity measurements of nanotube composites provide data that correlate with the formation of conductive paths. Measurements made on carbon fibres [9] and carbon nanotubes with high length-to-diameter aspect ratios indicate that loadings of only a few volume percent [10] are required to form conductive paths through the material compared to the high loading requirements of conductive carbon powders. Although permittivity measurements have been reported for epoxy composites of single-walled [11] and multi-walled [12] nanotubes, and for other resin systems [13, 14], these reports are often one off experiments that do not account for all the experimental variables, including feed stock purity, and may be difficult to reproduce.

In this report, the purity of several types of carbon nanotubes are measured and assessed in order to establish a baseline. First the purity of the MWNTs is determined by RAMAN spectroscopy, and thermogravimetric analysis (TGA). Different methods centred on sonication are studied for dispersing the nanotubes into an epoxy resin. Scanning electron microscopy (SEM) is used to investigate the dispersion of the carbon nanotubes through the resin matrix and permittivity measurements are made from 8 to 12 Gigahertz (GHz).

2 Experimental

2.1 Carbon nanotube stock materials

The carbon nanotubes used have different manufacturer reported diameters and lengths, Table 1. To facilitate dispersion into epoxy resin, some were functionalized with carboxylic acid groups. Also an epoxy resin-carbon nanotube master batch was acquired for comparison against in-house produced epoxy composites. Nanotube designations are:

- MWNT-COOH (for US Research Nanomaterials carboxylic acid functionalized),
- Cheap Tubes (for Cheap Tubes MWNT),
- Nanocyl-TWNT (for Nanocyl-7000 thin-walled nanotubes), and
- Epocyl (for an epoxy master batch part A, containing Nanocyl nanotubes).

In addition, catalyst, and any mesographitic and amorphous carbon, were removed from some of the Nanocyl-TWNT following a published purification method, where the nanotubes were ground, and then repeatedly refluxed in 3 M hydrochloric acid, followed by heating to 400 °C and higher [15]. These are designated as Purified. Purified Nanocyl tubes were acid functionalized by refluxing in aqueous 3 M nitric acid to add carboxylic acid functional groups, and are designated as Oxidized.

Table 1: Carbon nanotube sources and properties.

Supplier/ Designation	Details	Diameter (nm)	Length (μm)	Aspect ratio*
US Research Nanomaterials/ MWNT-COOH	MWNT, COOH functionalized (~2%) ~96% C purity, ~1.5% metal oxides.	10-20	10-30	1333
Cheap Tubes/ Cheap Tubes	MWNT.	30-50	10-20	375
Nanocyl/ Epocyl	Epocyl master batch NC E128-02, bisphenol A epoxy resin loaded with MWNT, loading approx. 3.3 wt%, cured with West System 206 hardener (5:1 A:B by weight).	9.5	1.5	158
Nanocyl/ Nanocyl-TWNT	Nanocyl-7000 thin-walled carbon nanotubes, Lot 257-1, industrial grade, ~90% C purity, ~10% Metal oxide.	9.5	1.5	158
Nanocyl/ Purified	Purified Nanocyl-7000 thin MWNTs were purified according to method by Huber [15].	9.5	1.5	158
Nanocyl/ Oxidized	Purified Nanocyl-7000 thin MWNTs are oxidized with nitric acid.	9.5	1.5	158

* Aspect ratios were calculated as length/diameter using averages of the manufacturers' reported length and diameter ranges.

2.2 Preparation of epoxy composites

Carbon nanotube composites were prepared using West System epoxy 105 part A and 206 part B in a 5:1 ratio by weight. The West System 206 Part B hardener was also used for curing the Epocyl epoxy part A resin, with the same 5:1 ratio. For Epocyl loadings below 3 weight percent, wt%, the resin was diluted with West System epoxy 105 part A before adding the hardener. Nanotube loadings ranged from 0.5 to 5 wt%. A typical composite preparation involved thinning the epoxy part A resin with 5-10 mL of ethanol or acetone. A weighed amount of carbon nanotubes were then hand mixed into the epoxy resin and sonicated for 30 minutes using a Branson 250 Digital Sonifier with the amplitude set to 60 watts. The nanotube-Part A resin was then heated to 90 °C to reduce the solvent. After the solvent was reduced and the resin cooled, the Part B epoxy hardener was added and a biplanetary mixer (Flacktek DAC 150 FVZ) was used to blend the Part A and Part B epoxy resins for 2 minutes at 1300 RPM. Resins were poured into an aluminum foil weighing pan and cured overnight or longer, at room temperature. Other methods to facilitate the dispersal of the carbon nanotubes involved the use of dispersants such as Disperbyk-2070, and BYK-9077, and by using carboxylic acid functionalized carbon nanotubes, such as the MWNT-COOH supplied by US Research Nanomaterials, and the Oxidized Nanocyl.

2.3 Material characterization

Samples of the nanotube-epoxy composites were fractured and the SEM was used to observe the distribution of carbon nanotubes through the material. The purity of the nanotubes was checked by TGA using a TA Instruments Q500. Nanotube samples were analysed in a pan open to air at a heating rate of 10 °C/min from room temperature to 1000 °C. RAMAN spectra of the carbon nanotubes were also used to check the purity and were obtained at three wavelengths, 532 nm, 638 nm and 785 nm, using a Horiba ExploRA RAMAN microscope. The nanotubes in Epocyl resin were isolated for analysis by diluting the resin with acetone and ethanol, centrifuging and decanting off the supernatant four times. Surface area measurements were made of Cheap Tubes, MWNT-COOH, and Nanocyl materials using a Quantachrome Nova 1200e instrument. Approximately 60 mg of nanotube material was weighed out for analysis and heated at 70 °C under vacuum for eight hours, then stored under vacuum until ready for use. These were further degassed for two hours at 70 °C and measurements were made in Helium mode, with N₂ adsorbate.

2.4 Permittivity measurements

The nanotube-epoxy composites were machined into rectangular blocks so as to fit into the inside of an X-band waveguide. The thickness of the samples was approximately 2 mm. Waveguide permittivity and permeability measurements were made over 8.2-12.4 GHz using an Agilent E8364c network analyzer to measure the S-Parameters. The network analyzer was calibrated using a full two-port Short-Open-Load-Thru (SOLT) calibration on the cables followed by a Thru-Reflect-Line (TRL) calibration of the waveguide. The permittivity and permeability were obtained by fitting calculated S-parameters to the measured values, where the calculated values are based on a simple slab model of the material. Uncertainties in the measurements are based on instrument resolution and are on the order of $\pm 5\%$ for the real component and $\pm 10\%$ for the lossy component, depending on the amount of nanotubes in the composite.

3 Results and discussion

3.1 Nanotube purity

Not all carbon nanotubes are made equal. The different processes, catalysts, feedstock, and post-synthesis treatments result in MWNT's having a range of diameters, lengths, and purity. Purity of the nanotubes is differentiated into the amount of residual catalyst, the amount of carbonaceous material such as amorphous and mesographitic carbon (porous graphite structures) [16], and the amount of carbon defects in the nanotubes themselves [17]. Knowledge of the amount and types of impurities in a nanotube supply may be important for explaining possible differences in the measured permittivity. Methods for characterizing carbon nanotube purity include transmission electron microscopy (TEM), SEM, TGA, RAMAN, and Fourier Transform Infrared Spectroscopy (FTIR) [17].

SEM and TEM imaging techniques provide visual indication of the MWNT's diameter, length, and entanglements, as well as the presence of amorphous and mesographitic carbon [16, 17]. FTIR has been used to provide information on the functional groups, such as carboxylic acids, attached to MWNT's, while RAMAN has been used to provide an estimation of the extent of defects in the nanotubes compared to the graphitic structure and long range order. The final technique, TGA, provides information regarding the amount of other carbonaceous species and residual catalyst.

3.1.1 FTIR

FTIR spectra of various carbon nanotubes were run using a Nicolet iS-50 FTIR with a Thunderdome Attenuated Total Reflectance accessory. The spectra collected using this method had peaks at about 1600 and 3500 cm^{-1} indicative of O-H, which have been observed for other nanotubes and has been thought to be due to trapped water [17]. The spectra for the MWNT-COOH and the Oxidized Nanocyl have additional peaks at about 1720 cm^{-1} indicative of carboxylic acid groups for these materials.

3.1.2 RAMAN

RAMAN spectra for the Cheap Tubes, Nanocyl-TWNT, and the MWNT-COOH are presented in Figure 1. These spectra have three major resonances at about 1340 cm^{-1} , designated as the disorder D-band, 1580 cm^{-1} , the graphitic G-band, and at 2600 cm^{-1} , the long range order G'-band, Table 2 [17, 18]. The ratios G'/D, D/G, and G'/G, of the measured intensity represented by these peaks have been related to the purity of the carbon nanotubes. The ratio G'/D is plotted for all the different types of MWNT's for each laser wavelength, Figure 2. The other band ratios, G'/G and D/G provide the same trends as observed in Figure 2, and the laser with the largest wavelength, lowest frequency, had the lowest sensitivity. Purity analysis by this method indicates that Cheap Tubes, MWCNT-COOH, and the rinsed Epocyl tubes with the epoxy resin removed were the purest and had the fewest defects. The Purified Nanocyl and Oxidized Nanocyl contained the greatest number of defects. This result is not unexpected as the steps taken to purify the Nanocyl and remove the catalyst involved a series of thermal oxidation and acid digestion treatments,

which likely created defects in the graphitic structure [15]. Note that amorphous and mesographitic material would reduce the intensity of the G and G' bands while increase the intensity of the D band.

Table 2: RAMAN vibrational modes of carbon nanotubes.

Peak Name	Frequency (cm ⁻¹)	Cause
D-band	~1350	Defects in the MWNT sample with sp ³ bonding and broken sp ² bonds in the sidewalls.
G-band	~1580	Crystalline/ graphitic nature of sample, tangential vibrations of the carbon atoms.
G'-band	~2700	Long range order in the sample arises from a two-phonon, second order scattering process that results in the creation of an inelastic phonon.

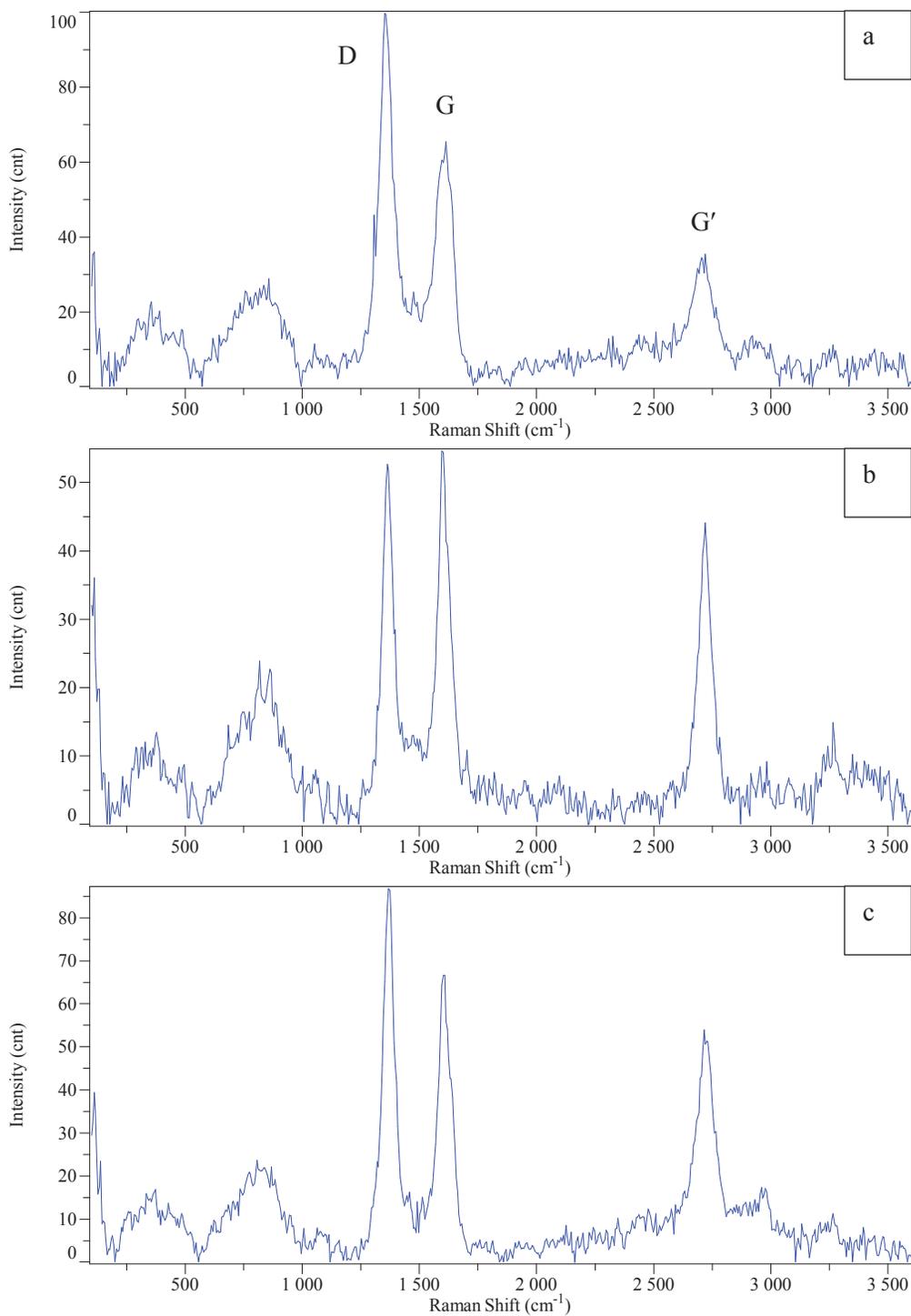


Figure 1: RAMAN spectra of (a) Nanocyl-TWNT, (b) Cheap Tubes, and (c) MWNT-COOH measured using a 532 nm laser. The D, G, and G'-bands are marked.

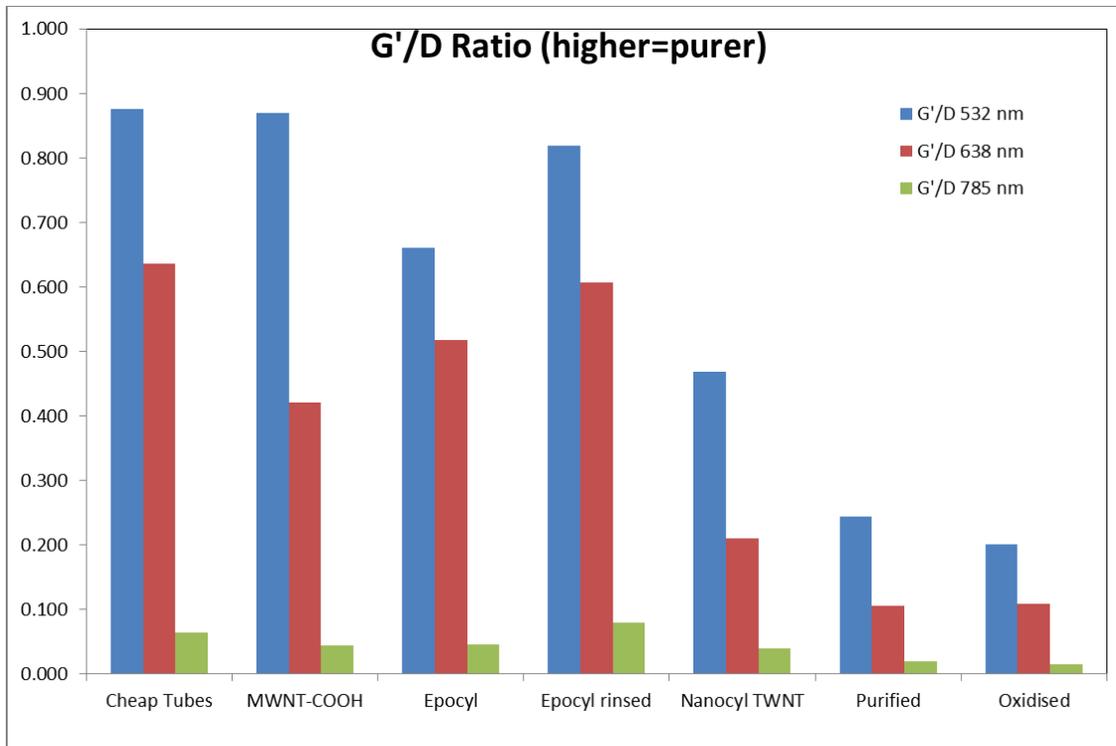


Figure 2: Ratio of the G'/D bands from the RAMAN spectra of the carbon nanotubes.

3.1.3 TGA

TGA monitors a sample's mass loss as it is heated in air. For carbon nanotube samples, mass losses can be indicative of the presence of amorphous carbon and graphitic particles which decompose at 200-300 °C, SWNT 350-500 °C, or MWNT's 400-650 °C [17]. Larger diameter and longer tubes result in higher oxidation temperatures. Residual mass after oxidation is attributed to the catalyst, which has been known to affect the oxidation temperatures of other species [17].

The decomposition temperature and purity of the carbon nanotubes was assessed using TGA, Figure 3. TGA measurements of Epocyl are not reported due to the presence of residual epoxy resin after it had been rinsed. The results indicate that there is only one thermal decomposition step for each sample, occurring between 400 and 650 °C. This is indicative of the nanotubes being fairly pure without large amounts of amorphous or mesographitic carbon being present. These impurities with defects and without the stabilized graphite structures would have oxidatively decomposed at lower temperatures than the nanotubes. The temperature at which the maximum rate of thermal decomposition occurred was lowest for the acid functionalized nanotubes, MWNT-COOH, and highest for the Cheap Tubes and the Purified Nanocyl. The unpurified Nanocyl-TWNT's fell between these two limits and had the largest amount of catalyst at 10.7%, Table 3. The catalyst should not significantly affect the permittivity as it was determined to be mainly alumina with less than 0.5% iron and cobalt content [15]. Purified Nanocyl had about 0.1 wt% residual catalyst. Cheap Tubes with the largest length and diameter,

and Purified Nanocyl have the highest oxidation temperature along with the lowest amount of catalyst. Carbon nanotubes that are long with large diameters, few defects and low levels of catalyst are expected to survive to higher oxidation temperatures which is consistent with these measurements [17]. Large diameter nanotubes have lower strain than those with smaller diameter, and longer tubes have fewer end defects per mass.

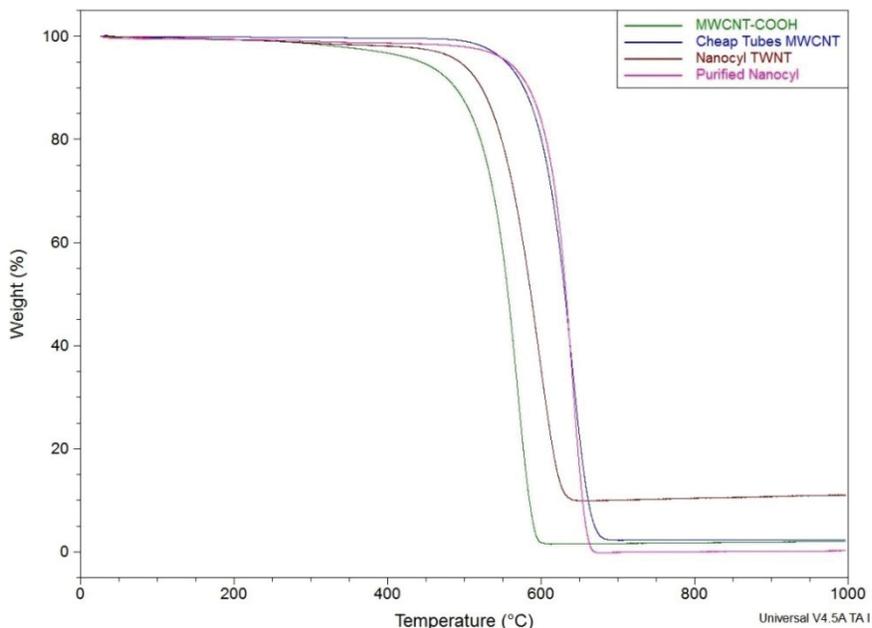


Figure 3: TGA runs of carbon nanotubes.

Table 3: Catalyst / non-combustible residue from TGA.

Sample	Residual (wt %)
MWNT-COOH	1.4
Cheap Tubes	2.4
Nanocyl-TWNT	10.7
Purified Nanocyl	0.1

3.2 Surface area measurements

Surface area measurements were made of Cheap Tubes, MWNT-COOH, and Nanocyl materials and found to be the lowest for Cheap Tubes and highest for Nanocyl, Table 4. The surface area of the average Cheap Tube is about four times as large that for Nanocyl, however, the mass per unit length of the average Cheap Tube is about 18 times as large as that for Nanocyl. This equates to a surface area for Nanocyl that is about 4 times as large as the surface area for the Cheap Tubes per unit mass. This trend is consistent with the observed surface areas. A consequence of using thinner nanotubes is that the electrical percolation threshold should be reached at lower loadings compared to thicker nanotubes.

Table 4: Nanotube surface area measurements.

Sample	Surface area (m ² /g)	Nanotube Diameter (nm)
Cheap Tubes	70	30-50
MWNT-COOH	105	10-20
Nanocyl-TWNT	270	9.5

3.3 Composite materials

The first step in making carbon nanotube-epoxy composites with reproducible properties is to homogeneously disperse the material into the epoxy resin. Strong cohesive forces and entanglements of the nanotubes, however, limit homogeneous dispersion unless the tubes can be separated. Methods have been developed for the dispersion of nanotubes including ultrasonication which was used in this work [7]. Ultrasonic dispersion of the carbon nanotubes into the epoxy resin, diluted with ethanol or acetone, visibly reduced the size of agglomerated material in the epoxy resin. Two commercial dispersants and carboxylic acid functionalized nanotubes were trialed to see if they improved nanotube dispersion in the epoxy resin. This is discussed further with respect to the permittivity results below.

3.4 SEM

SEM was used to assess how well the carbon nanotubes were distributed through the epoxy resin, Figure 4. To make these observations, nanotube-epoxy composites were fractured by snapping the composite, and the fracture surface imaged. In a couple of cases the composites were first frozen in liquid nitrogen before being snapped. A light gold coating of the surface generally improved the ability to image the nanotubes. For the composites made with the short Nanocyl-TWNT, including the Epocyl, Purified, and Oxidized materials, the carbon nanotubes appeared to be fairly well distributed. Examples of this are seen in Figures 4a and b, for 5 wt% Nanocyl-TWNT and 2.5 wt% Epocyl, respectively. On the other hand, composites made with the longer nanotubes such as MWNT-COOH and Cheap Tubes, still contained large nanotube clusters, Figure 4c. These clusters are seen as large light patches. These observations indicate that the shorter Nanocyl-TWNT nanotubes are more easily separated from aggregates than the much longer nanotubes, and that longer or more aggressive processing methods are required to disperse the longer nanotubes. A method for monitoring the extent of de-aggregation is required, such as determining viscosity or particle size.

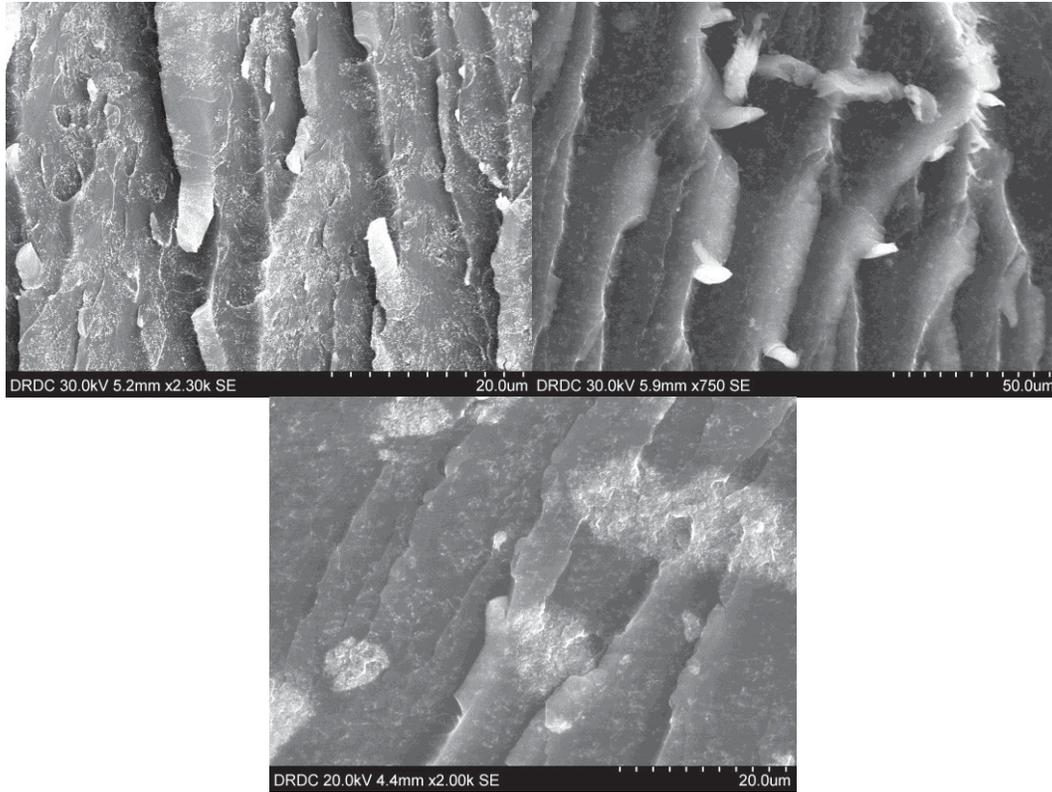


Figure 4: SEM images of the fracture surfaces of carbon nanotube-epoxy composites: a) 5 wt% Nanocyl-TWNT, b) 2.5 wt% Epocyl, and c) 5wt% MWNT-COOH.

3.5 Permittivity

The carbon nanotube-epoxy composites were machined into rectangular blocks with dimensions suitable for fitting into a section of X-band waveguide and the S-Parameters were measured for the microwaves reflecting from, and transmitting through the sample. The real, ϵ_r' , and imaginary, ϵ_r'' , components of the relative permittivity were determined by fitting calculated S-Parameters to the measured ones, using a homogeneous one layer model of the carbon nanotube-epoxy composite. The real and imaginary components of the relative permittivity are plotted in Figures 5 and 6 for four types of carbon nanotubes.

The permittivity of the Nanocyl-TWNT was significantly larger than the permittivity measured for the other nanotube-epoxy composites. The permittivity for Epocyl and MWNT-COOH were very similar to each other and Purified Nanocyl had permittivity values that were slightly higher. Oxidised Purified Nanocyl did not have permittivity values significantly different from those of the Purified material. The use of dispersants did not affect the measured permittivity.

The aspect ratio of the carbon nanotubes should directly correlate to the measured permittivity with long narrow nanotubes being able to form conductive networks through the material at lower loading than high aspect ratio nanotubes. However, this was not observed with Nanocyl-TWNT,

which has the smallest aspect ratio of the materials studied, and the highest permittivity measured. This probably stems from two properties of this nanotube source: the short length of the nanotubes on the order of 1.5 μm , and the narrow diameter of the tubes, about 9.5 nm. The short length of the nanotubes appears to have made it easier to de-aggregate and disperse them through the matrix. The narrow diameter of the Nanocyl means that on a weight-for-weight basis, there will be a greater number density, or overall length of nanotubes in the matrix compared to nanotubes with thicker diameters, assuming that both materials have the same density. The combination of these two factors indicate that Nanocyl-TWNT should reach the percolation threshold sooner, and have higher conductivity and permittivity than the longer, larger diameter material, as was observed in the measurements. As mentioned in section 3.1.3, the catalyst content of the Nanocyl-TWNT was mainly alumina with less than 0.5% iron and cobalt which would not significantly affect the permittivity and permeability [15]. As to why the Purified Nanocyl has lower permittivity, it was observed in the RAMAN spectroscopy of these materials that the purification process removed catalyst, however, it introduced defects into the material which would lower its conductivity and hence permittivity.

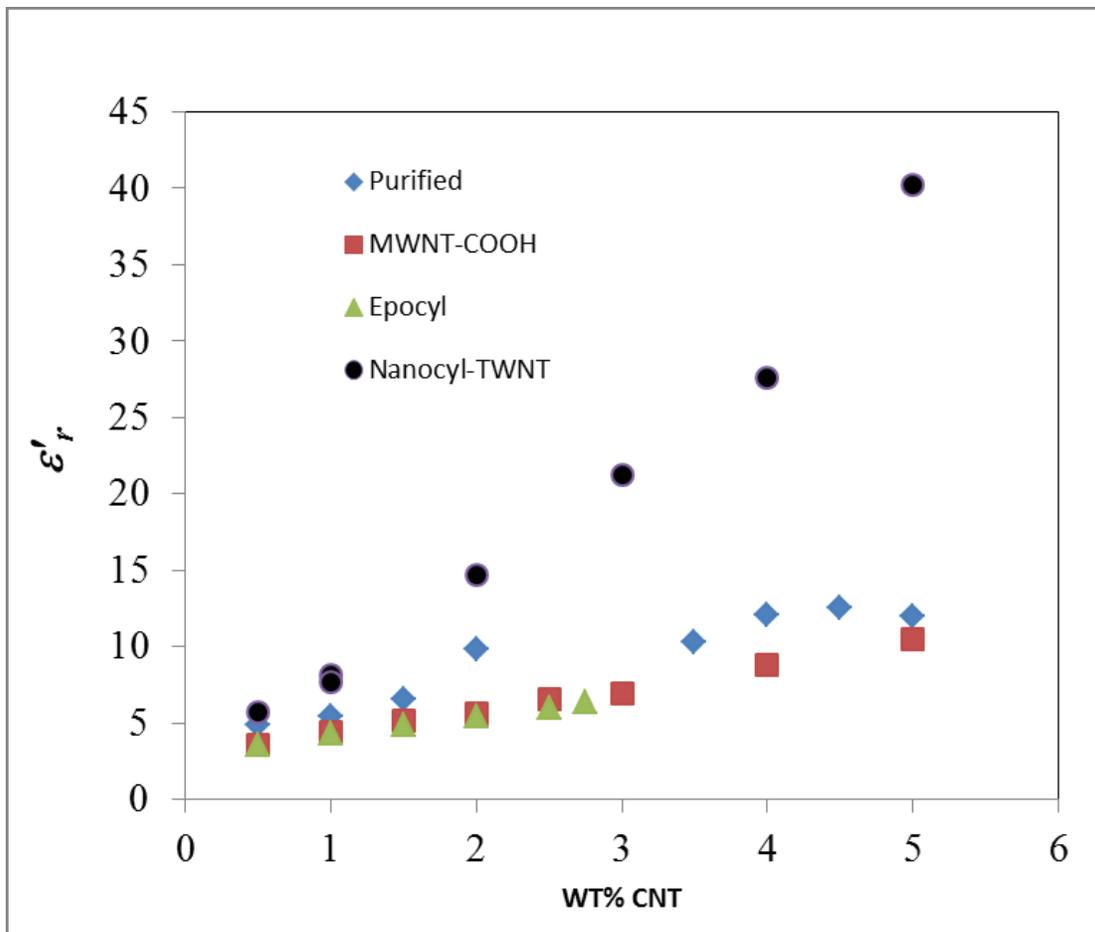


Figure 5: Real component of the relative permittivity as a function of the nanotube loading measured at 10 GHz.

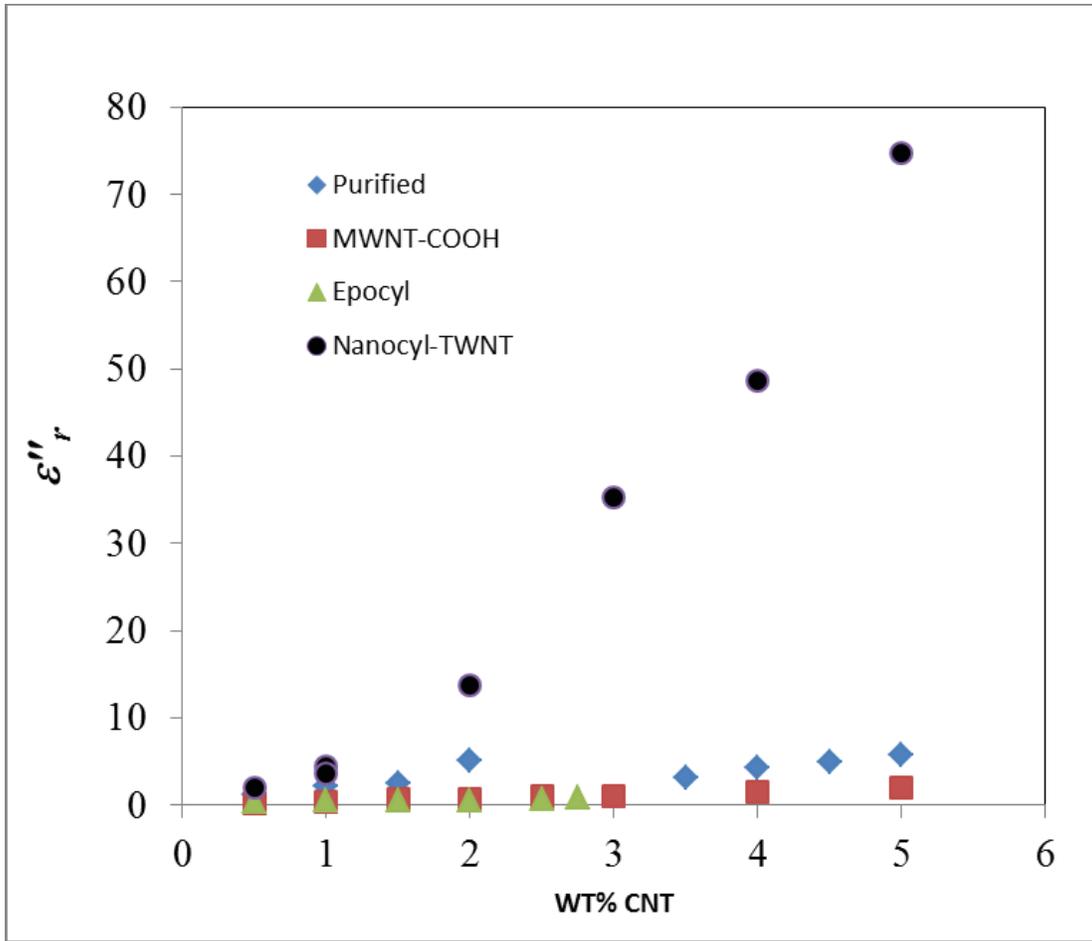


Figure 6: Imaginary component of the relative permittivity as a function of the nanotube loading measured at 10 GHz.

4 Conclusions

A series of methods for analyzing the quality of carbon nanotubes and their epoxy composites have been presented. FTIR provides evidence of nanotube surface functional groups. RAMAN spectroscopy is useful for determining the chemical defects in the material, while TGA provides indication of the presence of catalyst and other carbon forms. SEM was useful in providing an indication of the de-aggregation of the nanotubes and their dispersion through the epoxy matrix, while the permittivity measurements are indicative of the nanotube connectivity within the material.

In terms of providing a baseline for the materials studied, Cheap Tubes and MWNT-COOH appear to have fewer defects than the Nanocyl-TWNT based materials. Cheap Tubes had the most thermal stability, and MWNT-COOH the least. The purification and oxidization processes used to achieve Purified and Oxidized material introduced defects into the nanotubes. The two most important factors for dispersing the nanotubes into the epoxy for achieving high permittivity appear to be having short nanotubes that are not as entangled and narrow diameters ensuring more overall tube length in the composite.

Finally a process for monitoring the solution viscosity or nanotube particle size should enable reproducibility in obtaining material properties.

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List of symbols/abbreviations/acronyms/initialisms

DRDC	Defence Research and Development Canada
FTIR	Fourier transform infrared
GHz	Gigahertz
MWNT	Multi-walled carbon nanotube
MWNT-COOH	US Research Nanomaterials product designation MWNT-COOH
SEM	Scanning electron microscope
RAMAN	RAMAN is spectroscopic technique. The spectra arising from RAMAN spectroscopic measurements may be referred to as RAMAN spectra. RAMAN spectroscopy may be referred to as RAMAN.
SOLT	Short-Open-Load-Thru, a waveguide calibration method
sp^2	Refers to hybridization of s and p orbitals to form pi-bonds
sp^3	Refers to hybridization of s and p orbitals to form C-H single bonds
SWNT	Single-walled carbon nanotube
TGA	Thermogravimetric analysis
TRL	Thru-Reflect-Line, a wave guide calibration method
wt%	Weight percent

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The mechanical and electrical properties of carbon nanotube composites are attractive for a number of applications. The final properties depend on the quality of the feed stock, the amount of material used, and methods for de-aggregating and dispersing the nanotubes. Here, Fourier transform infrared spectroscopy, RAMAN spectroscopy, scanning electron microscopy, thermogravimetric analysis, and permittivity measurements are used to discern between three commercially available multi-walled carbon nanotubes (Nanocyl 7000, Cheap Tubes, and a sample of acid functionalized multi-walled nanotubes), and their epoxy composites. The RAMAN and thermogravimetric analysis data indicated that the Nanocyl had the most defects and impurities, however, the permittivity measurements and scanning electron microscopy data indicated that the Nanocyl nanotubes were well distributed in the matrix. The most important factors for dispersion and high permittivity appear to be the ability to de-aggregate the nanotubes, which is enabled by the short length, and a small diameter. The small diameter results in an overall longer length of carbon nanotube in the composite compared to an equal mass of larger diameter nanotubes.

Les propriétés mécaniques et électriques des matériaux composites à base de nanotubes de carbone sont intéressantes pour un certain nombre d'applications. Les propriétés finales de ces matériaux dépendent de la qualité des matières premières, de la quantité utilisée et des méthodes de désagrégation et de dispersion des nanotubes. Pour le présent travail, nous avons utilisé la spectroscopie infrarouge à transformée de Fourier, la spectroscopie RAMAN, la microscopie électronique à balayage, l'analyse thermogravimétrique et des mesures de permittivité pour faire la distinction entre trois nanotubes de carbone multiparois disponibles sur le marché, (Nanocyl 7000, Cheap Tubes et un échantillon de nanotubes de carbone multiparois à fonctionnalité acide) et leurs composites époxydiques. Les données des analyses RAMAN et thermogravimétrique ont montré que le Nanocyl comportait le plus grand nombre de défauts et d'impuretés. Cependant, les mesures de permittivité et les données de la microscopie électronique à balayage ont indiqué que les nanotubes Nanocyl étaient bien répartis dans la matrice. Les facteurs de dispersion et de permittivité élevée les plus importants semblent être la capacité de désagrégation des nanotubes, qui est activée par une courte longueur et un petit diamètre. Le petit diamètre entraîne une plus grande longueur globale des nanotubes dans le composite comparativement à une masse égale de nanotubes de plus grand diamètre.

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Carbon Nanotubes; Multi-walled Nanotubes; Permittivity; Microwave Absorbers; Epoxy Composite