

Enhanced electromagnetic interference shielding through the use of functionalized carbon nanotube-reactive polymer composites

Sung-Hoon Park, Paul Theilmann, *Student member, IEEE*, Peter Asbeck, *Fellow, IEEE* and Prabhakar R. Bandaru

Abstract— We report on a new principle yielding enhanced electromagnetic shielding, using as an example, a composite comprised of carbon nanotubes (CNT) integrated with a Reactive Ethylene Terpolymer (RET). Such composites were synthesized through the chemical reaction of the functional groups on the CNT with the epoxy linkage of the RET polymer. The main advantages of these composites incorporate good dispersion with low electrical percolation volume fractions (~0.1 volume %), yielding outstanding microwave shielding efficiency for EMI applications. The shielding effectiveness was characterized for both single-walled and multi-walled CNT based composites and was much enhanced in the former. The specific roles of absorption and reflection in determining the total shielding, as a function of the nanotube filling fraction, is also discussed.

Index Terms— Carbon nanotubes, polymer composites electromagnetic interference shielding, functionalization

I. INTRODUCTION

THE advent of carbon nanotubes (CNTs) and recent studies in polymer-CNT composites[1] has motivated study of their use for Electromagnetic Interference (EMI) applications as light-weight, high shielding efficiency materials. At the very outset, in EMI shielding, the effects of both reflection and absorption of the incident EM radiation have to be considered[2]. The intensity of the reflected radiation (R) is related to the difference of the characteristic impedances (Z) of the incident medium (i.e., air of impedance Z_a) and the material (Z_m) through $R = \left(\frac{Z_a - Z_m}{Z_a + Z_m}\right)^2$. The absorption (A) is

quantified through the skin depth (δ), the extent to which the radiation, of frequency f , penetrates the material, through $\delta = \sqrt{\frac{1}{\pi f \mu \sigma}}$, where μ is the magnetic permeability and

σ the electrical conductivity. Consequently, while metallic materials (with low Z_m and high σ) are obviously efficient shielding materials, issues such as cost and bulk preclude their wide scale application. Such issues have encouraged the use of polymers where the intrinsic poor conductivity could be compensated through the use of filler materials which form a conducting network. (It is to be noted that electrically conducting polymers are currently expensive, difficult to process, and need considerable improvement in their mechanical properties[3]).

While carbon based materials, such as carbon black and fibers, have previously been used in polymer composites[4], their widespread use is limited due to a maximum loading capability, beyond which there is embrittlement. CNTs are then more attractive candidates for filler materials in composites, primarily due to their large aspect ratio and tunable electrical conductivity, which enables electrical percolation to be achieved with *very small* amounts of nanotubes. For example, we have calculated from the excluded-volume percolation theory of rod-like systems [5, 6] (Figure 1a) that single walled CNTs (SWNTs) with an aspect ratio of ~ 5000 (which corresponds to a length of $5 \mu\text{m}$ and a diameter of $\sim 1 \text{nm}$), when dispersed uniformly into a non-conducting polymer enable a conducting pathway at a volume fraction of $\sim 0.01 \%$. Our results on SWNT-RET polymers, which we report in this paper, yield values very close to the optimal considerations (as indicated by the \star in Fig. 1a).

In our experiments, we have also observed higher values of the EMI shielding efficiency (SE) at a lower loading fraction of SWNTs (i.e., $\sim 30 \text{dB}$ at $4.5 \text{vol } \%$ of functionalized SWNTs) compared with literature. A few examples of the SE values from literature include: (i) $< 17 \text{dB}$ with $15 \text{vol } \%$ SWNTs in SWNT/epoxy composites[7, 8] (ii) $< 20 \text{dB}$ with $11.6 \text{vol } \%$ SWNTs in SWNT/polyurethane composites at 8GHz [9], (iii) $\sim 20 \text{dB}$ with $8 \text{vol } \%$ SWNTs in SWNT/vinyl acetate based composites at 8GHz [10]. The superior performance of our particular composites could possibly be due to the uniformity of dispersion obtained through functionalization along with a larger aspect ratio. Additionally, our values are comparable to those obtained in CNT films, of $\sim 28 \text{dB}$ at 10GHz [11]

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S. H. Park and P. R. Bandaru are with the Materials Science Program, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093, USA. (e-mail: shp003@ucsd.edu; pbandaru@ucsd.edu).

P. Theilmann and P. Asbeck are with the Department of Electrical and Computer Engineering, University of California, San Diego, La Jolla, CA 92093, USA. (e-mail: ptheilma@ucsd.edu; asbeck@ece.ucsd.edu).

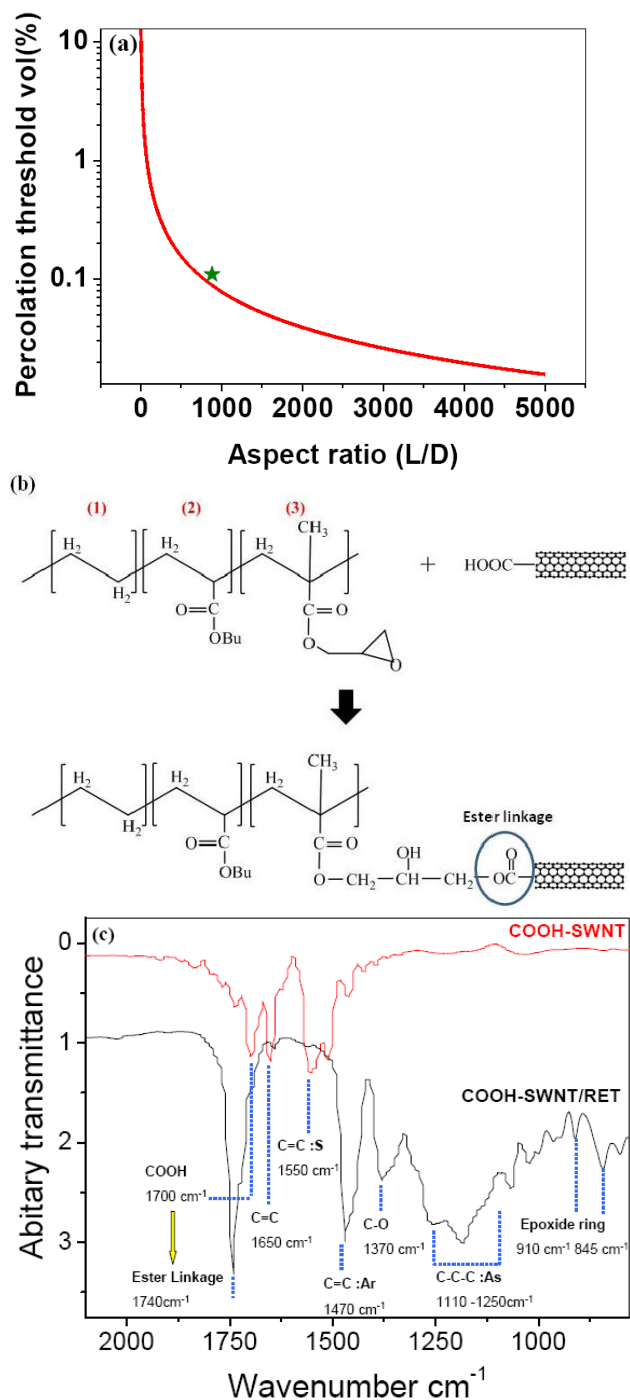


Fig. 1. (a) A high aspect ratio, pertinent to carbon nanotubes, enables very low threshold volume fractions for electrical percolation. The threshold-aspect ratio curve, for uniformly dispersed nanotubes, has been simulated from percolation theory where the * indicates results from our experimental measurements.

(b) Schematic diagram of the reaction between functional groups on the CNT with the epoxy groups of the Reactive Ethylene Terpolymer (RET) constituted from (1) polyethylene, (2) methyl-methacrylate, and (3) epoxide functional groups. While (1) and (2) contributes to the mechanical robustness, (3) is used for forming ester linkages to the $-\text{COOH}$ groups on the functionalized nanotubes for enhanced bonding and dispersion.

(c) Fourier Transform Infrared (FT-IR) Spectrographs comparing the (i) bare $-\text{COOH}$ functionalized SWNT (COOH-SWNT) with the (ii) functionalized single walled CNT-RET (COOH-SWNT/RET) composites. The evidence of esterification, and bonding, between the functionalized nanotubes and the RET is seen in the energy up shift of the 1700 cm^{-1} peak to 1740 cm^{-1} (Ar: Aromatic, S: Symmetric, As: Antisymmetric)

II. EXPERIMENTAL PROCEDURE

For large scale processing, it is desirable[1] to ensure effective *mixing* and dispersion of the CNTs within the polymer. The consequent enhanced nanotube-polymer interactions which are obtained, for example, through chemical functionalization, enable low critical volumes for electrical percolation. In this paper, we tackle such issues in detail and also consider the relative influences of both single walled and multi-walled carbon nanotubes in terms of their shielding efficiency.

We chose for our experiments, a composite of CNTs and a Reactive Ethylene Terpolymer (RET: Elvaloy 4170, density 0.94 g cm^{-3}) constituted from (1) polyethylene, (2) a polar methyl-methacrylate group, and (3) epoxide functional groups – Fig. 1b. While (1) and (2) contribute to the mechanical characteristics (e.g., elastomeric properties) and corrosion resistance underlying the utility of RET as a hot-melt adhesive and coating, the epoxy group has high reactivity[12] and is amenable for effective anchoring of the ring bonds with functional groups(e.g. $-\text{OH}$, COOH , $-\text{NH}_2$ etc.) on the CNTs. The underlying rationale is that the epoxide ring rupture [13] on the RET would be facilitated by the $-\text{COOH}$ groups on the functionalized nanotubes (Figure 1b) and then contribute to bonding of the COOH on the SWNT with the epoxy group of the RET.

The effects of both pristine and carboxyl functionalized SWNTs (average diameter 1-2 nm, length 5-20 μm , 90% purity, and density 2.1 g cm^{-3}) and multi-walled carbon nanotubes (MWNTs, average diameter 140 nm, length 5-9 μm , 90% purity, and density 1.7 g cm^{-3}) were examined. A mixture of sulfuric and nitric acids (in a 3:1 ratio) was used both for nanotube surface functionalization, with $-\text{COOH}$ groups, and for removing impurities[12]. Subsequently, the CNTs were rinsed with deionized water, and then dried at $60 \text{ }^\circ\text{C}$ for 10 hours. The MWNTs and SWNTs were then dispersed in toluene with sonication for 20 minutes. (It was typically seen, through atomic force microscopy characterization that the above procedure, including sonication, reduces the average length of the SWNTs to $\sim 4.3 \mu\text{m}$, with a bundle diameter $\sim 4.8 \text{ nm}$, resulting in an aspect ratio of ~ 880 . On the other hand, the MWNTs have an average length of $5.6 \mu\text{m}$, with a bundle diameter $\sim 190 \text{ nm}$, yielding an aspect ratio of ~ 30). The RET was then added to toluene solvent with heating to $\sim 60 \text{ }^\circ\text{C}$ for 2 hours and subsequent stirring. The CNT dispersion was then added to the RET solution and sonicated again for ~ 50 minutes. To remove excess solvent, the mixture was stirred, at $60 \text{ }^\circ\text{C}$ for 3 hours, poured into glass dishes and then evacuated in vacuum (10^{-3} Torr) for 12 hours. Subsequently, a hot press was used to fabricate the composites into desired thickness.

Fourier Transform Infrared (FT-IR) Spectroscopy (Nicolet Magna-IRTM Spectrometer 550) was used to provide proof of covalent bonding [14] between the CNTs and the RET as evidenced by the energy up-shift of the $\text{C}=\text{O}$ stretch (at 1700 cm^{-1}) - due to the carboxyl group of the $-\text{COOH}$ functionalized SWNT- to 1740 cm^{-1} due to the formation of an ester linkage (Fig. 1c). The exact location of the functional groups would depend on the defect density on the nanotubes and can be manipulated[15].

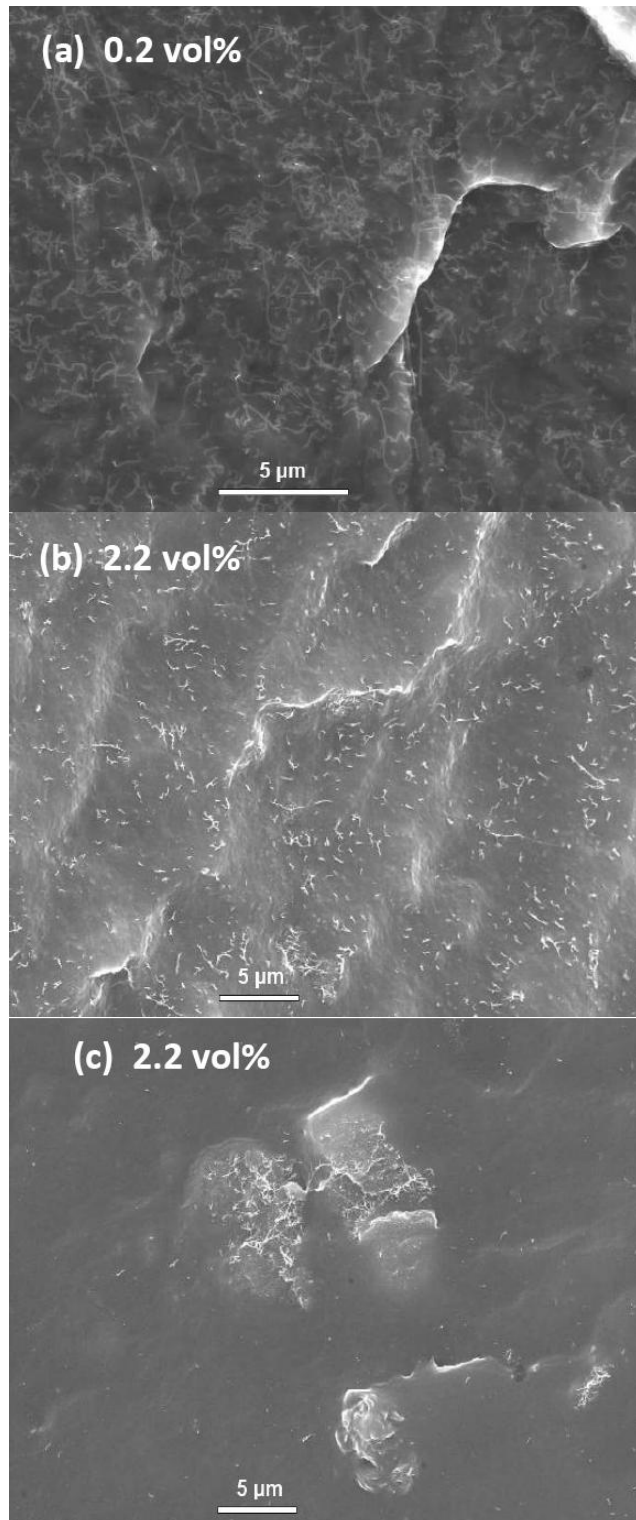


Fig.2. Uniform dispersion of functionalized SWNTs in the RET polymer is indicated, through Scanning Electron Microscope (SEM) micrographs, at both (a) 0.2 volume % and (b) 2.2 volume % filling fractions of the SWNTs. On the other hand, (c) non-functionalized SWNTs dispersed into the RET matrix exhibit clumping.

However, if the defects are considered to be randomly dispersed, isotropic bonding of the CNTs with the polymer matrix is implied yielding uniform dispersion and mixing. While different strategies have been used for homogeneous dispersion[16, 17], it has been generally observed that covalent

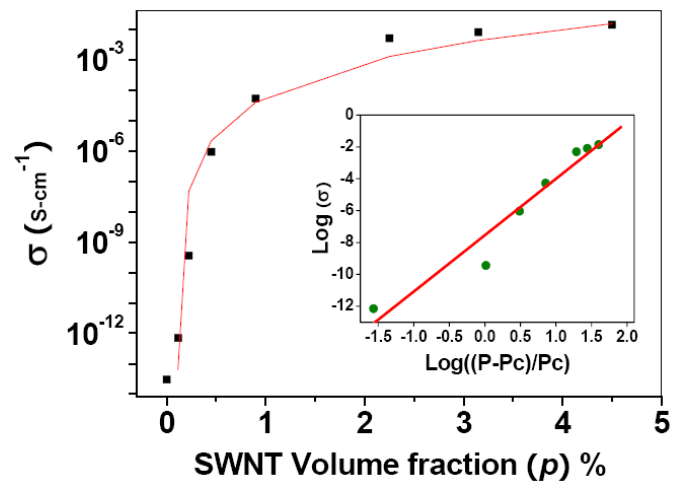


Fig.3. The electrical conductivity of the functionalized SWNT-RET composites follows a power law characteristic of percolation like behavior. The inset shows log-log plot of σ vs. $(\rho - \rho_c)/\rho_c$, exhibiting a threshold volume fraction, $\rho_c \sim 0.11\%$ and a slope ~ 3.4 .

stabilization [18] and intimate chemical reaction[14, 19] could be beneficial for enhanced electrical conductivity and electromagnetic shielding effectiveness. In line with the above reasoning, we have indeed observed a more uniform dispersion using functionalized SWNTs for both low (Fig. 2a) and high (Fig. 2b) CNT filling fractions, as observed through the Scanning Electron Microscopy (SEM: Phillips XL30) images of the fracture surfaces of the CNT/RET composite. The beneficial effects of our proposed functionalization scheme are also evident through a more homogeneous dispersion of the CNTs in the polymer matrix in comparison to the clumping observed (Fig. 2c) when un-functionalized CNTs are mixed into the polymer. (Note that more rigorous quantification of uniform dispersion is challenging due to the different length scales involved[20]). A thickness of ~ 2 mm of the CNT-RET polymer composite was used for all measurements reported in this paper.

The composites were treated with oxygen plasma (Oxford Plasmalab 80 RIE) prior to electrical contacting of the surface. Subsequently, 50nm of gold were sputtered on for the contacts. The four-wire resistance method was used to measure the resistance (R) for composites with $R < 1G \Omega$, using the Keithley 487 picoammeter and the Keithley 2400 Sourcemeter. For such measurements, we used samples with rectangular cross-section (5 mm wide X 2 mm thick, typically). The outer current leads were separated by 25 mm while the inner voltage leads were separated by 15 mm. For higher resistance ($> 1G \Omega$) composites, two point measurements using the Agilent B1500A semiconductor device analyzer, with triaxial probes were used.

We next monitored the SE of the synthesized composite in the microwave frequency range (8.2-12.4 GHz: X-band) using a two-port vector network analyzer (Agilent 5242A PNA-X). The X-band is used for both civil and military communications with applications as diverse as weather monitoring, vehicular detection and air traffic control and defense tracking[21]. The desired amount of conducting filler is generally dependent on

the required SE, where $SE = 10 \log \frac{P_i}{P_t}$, where P_i and P_t are the magnitudes of the incident and transmitted power densities and

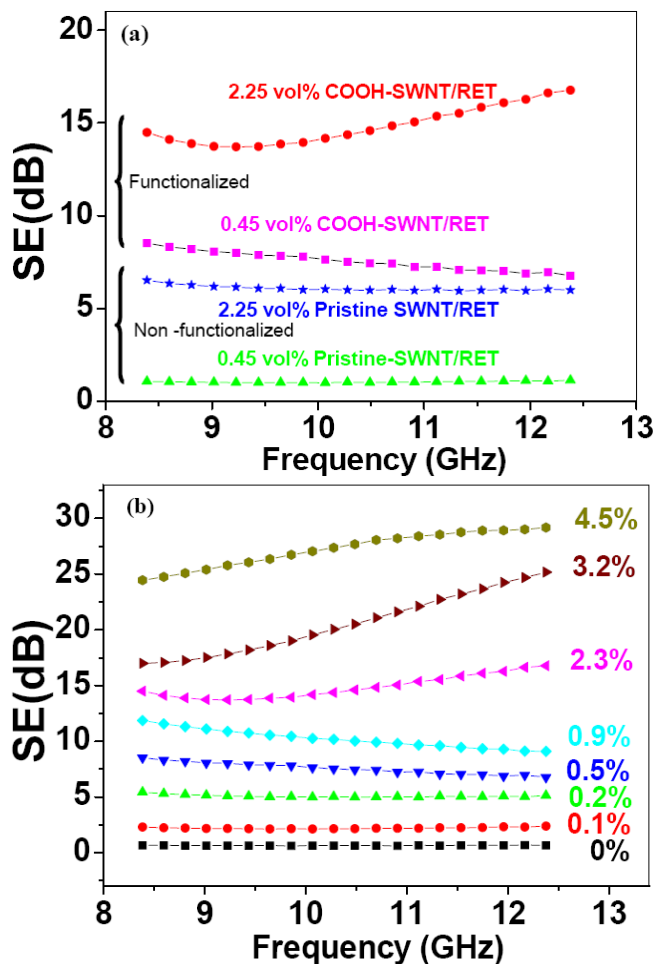


Fig.4. (a) An enhanced shielding effectiveness (SE) has been observed for the chemical functional group reaction enhanced SWNT-RET polymer composites(COOH-SWNT/RET) compared to non-functionalized SWNT-RET composites (Pristine SWNT/RET), (b) The SE increases with increased filling fraction (depicted in %)of the SWNTs

were measured through the network analyzer. The initial calibration was performed using the TRL (Thru-Reflect-Line) method [22] which is an improvement over the traditional OSL (Open-Short-Load) type calibration techniques. Using the TRL method, the “Thru” measurement was first performed by connecting the two 15 cm waveguide sections directly and measures the total loss and phase delay of the setup, in the absence of the sample. For the “Reflect” calibration, an aluminum short/load was placed between the waveguide sections, while the “Line” calibration was done through the insertion of a quarter-wave section of waveguide (WR-90). The latter measurements accurately delineate the plane of the DUT (device under test) through a consideration of the reflections and other losses prevalent in the test setup including the cabling, waveguide segments, and transitions, in the absence of a sample. The scattering parameters obtained from the calibration were then used for the baseline.

The CNT-polymer composite was pressed into a sample holder of size 0.9” X 0.4” - standard size for the X-band frequency range. The composite loaded holder was inserted between two 15 cm lengths of WR-90 X-band waveguide to

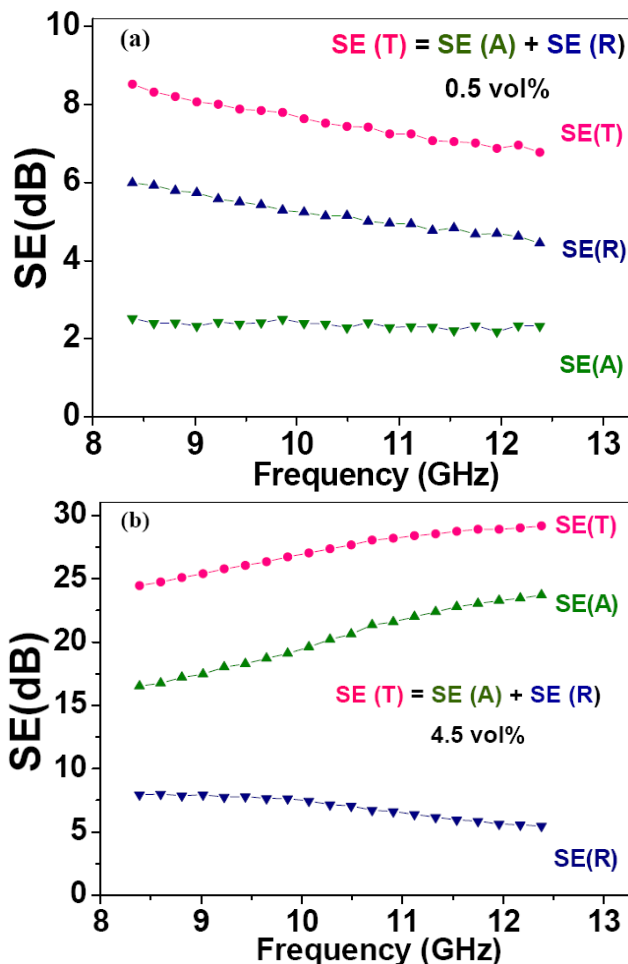


Fig.5. The total shielding efficiency, SE (Tot), can be decomposed into contributions from reflection, SE (R) and absorption, SE (A) through a consideration of the frequency dependence. (a) Reflective losses dominate the total SE for low (e.g., ~ 0.5 volume %) nanotube filling fractions, while (b) the absorption is more important at larger (e.g., ~ 4.5 volume %) fractions.

mitigate the near-field effects of the coax to waveguide transitions. It should be noted that while the measured SE, in our experiment, is not identical to the plane wave, free space SE due to the confinement and wave polarization effects[23], it can be shown that the difference is negligible[8, 10].

III. RESULTS AND DISCUSSION

We observed through DC electrical conductivity (σ) measurements on the SWNT-RET composite electrical percolation like behavior (Figure 3), which could be fit to a power law of the form: $\sigma \sim \sigma_0(p - p_c)^\beta$, where p_c is the percolation threshold volume fraction (p) and β a critical exponent. σ_0 is a constant for a particular filler-polymer combination. The obtained values of p_c (~0.11%) and β (~3.4) from the best fit of the conductivity data to the log-log plots of the power laws in the inset to Fig. 3 are appropriate for a thick film resistor configuration with elongated fibers[24]. It should be noted that at higher frequencies, capacitive coupling,

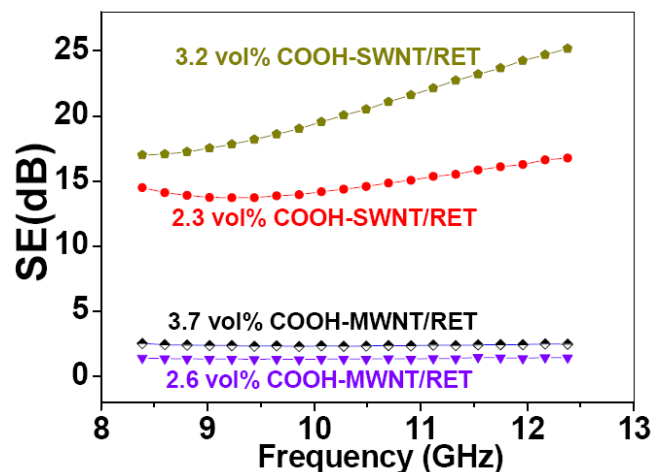


Fig.6. The SE is generally larger for the SWNT based composites compared to the MWNT based composites and is indicative of the influence of the aspect ratio (SWNT ~ 880, MWNT ~ 30) in determining the percolation and the SE.

in addition to continuous electrical paths between the CNTs can contribute significantly to the electrical conductivity [25].

We noticed that an order of magnitude higher SE was obtained for the COOH- functionalized CNTs compared to a dispersion of non-functionalized SWNTs in the RET polymer matrix (Fig. 4a). Such an effect is presumably due to the effect of uniform dispersion resulting in homogeneous connectivity of the SWNT fillers. As expected, the SE increases with increasing amount of SWNT filler (Fig. 4b).

Probing into the dependence of the SE vs. frequency, we notice (e.g., Fig. 4), that at low (/high) CNT filling fractions the SE decreases (/increases) with frequency while exhibiting a minimum at intermediate filling fractions (e.g., ~ 2.3 volume % - Figure 4b). One can understand such variations by considering the total effective shielding effectiveness, $SE(Tot)$, of the composite as the sum of the contributions due to both the reflective (R) and the absorptive (A) components, through $SE(Tot) = SE(R) + SE(A)$, where $SE(R) = -10 \log(1-R)$ and $SE(A) = -10 \log\left[\frac{T}{1-R}\right]$ [21, 26]. The R, A, and the T

(transmission) were then obtained through the determination of the S-parameters using the vector network analyzer, where $T = |S_{21}|^2$, $R = |S_{11}|^2$, and $A = 1 - |S_{11}|^2 - |S_{21}|^2$. It was assumed that the effect of multiple reflections on the SE is negligible for our considered composite thickness [4, 7]. It was then seen that the $SE(R) > SE(A)$ for low volume % nanotube filling fractions – Fig. 5a, while $SE(A) > SE(R)$ for higher filling fractions – Fig. 5b. It could be inferred that the cross-over from reflection dominated shielding to absorption dominated shielding occurs ~ 2.3 volume % (as seen in Fig. 4b). A preliminary explanation, and fit to experiment, where $SE(R)$ decreases with frequency (f) while $SE(A)$ increases with f was obtained through examining the form of R which varies as ~

$$\log_{10}\left(\frac{\sigma}{f}\right)$$

through its dependence on the reflection loss and A, which is thickness (t) dependent, and varies as $t\sqrt{f\sigma}$ [27]. Simulations and experiments are in progress to explore such phenomena in more detail.

Interestingly, the SE is also much larger, almost by a factor of 100, for functionalized SWNTs compared to functionalized MWNT – RET composites (Fig. 6). We have noted previously (Section II) that the aspect ratio of the MWNTs are smaller than the SWNTs, which implies (from Fig. 1a) reduced percolation and lower SE. We have experimentally observed that several variables such as (1) the aspect ratio of the CNTs, (2) DC and AC electrical conductivity of SWNTs/MWNTs, and (3) the thickness of the composite, in addition to (4) frequency, can be used as knobs to determine practical applicability of the CNT-RET polymer. One example, as we have shown, is the way in which nanotube type and/or concentration modulates the reflective or absorptive components of EMI shielding.

IV. CONCLUSIONS

Our study has provided an example of a nanotube (SWNT/MWNT)-polymer (e.g., RET) system which utilizes functional group interactions for achieving uniform dispersion with low electrical percolation concentrations and enhanced microwave shielding effectiveness. Analysis of the total observed EMI shielding was used for investigating the mechanisms of absorption and reflection in the composite, where a cross-over from reflection dominated shielding to absorption dominated shielding was observed at a CNT volume fraction of ~ 2.3%. It was also concluded that SWNTs were much more effective compared to MWNTs. Preliminary experiments probing the mechanical characteristics of the proposed CNT-RET polymer composites have indicated increased tensile strength as a function of increasing volume fraction of the CNTs. In summary, our proposed scheme of using functionalized CNT-polymer composites together with our obtained results could be used as a basis for light-weight, high shielding efficiency materials for EMI applications.

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Sung-Hoon Park received the B.S degree in mechanical engineering from Chung-Ang University, Seoul, Korea, in 2002, M.S. degree in mechanical and aerospace engineering from University of California, San Diego (UCSD) in 2006, and is currently working toward the Ph.D. degree in materials science program, department of mechanical and aerospace engineering at UCSD. His research fields are synthesis of functionalized nanocomposites (carbon nanotube, nanocoil, nanowire) and thin film for electromagnetic interference (EMI) shielding material, microwave characterization of nanocomposites and nanopatterned surface.



Paul T. Theilmann (S'08) received the B.S. and M.S. degrees in electrical engineering from the University of California at San Diego (UCSD), La Jolla, in 2004 and 2006, respectively, and is currently working toward the Ph.D. degree at UCSD. During his M.S. studies he designed a low loss, integrated phase shifter for the linearization of a highly efficient Doherty power amplifier. His research interests include integrated circuits for wireless communications and wireless biomedical implants.



Peter M. Asbeck (M'75-SM'97-F'00) attended MIT, where he received the B.S. and Ph.D. degrees in 1969 and in 1975. He worked at the Sarnoff Research Center, Princeton, New Jersey, and at Philips Laboratory, Briarcliff Manor, New York, in the areas of quantum electronics and GaAlAs/GaAs laser physics. In 1978, he joined Rockwell International Science Center, where he was involved in the development of high speed devices and circuits using III-V compounds and heterojunctions. He pioneered efforts to develop heterojunction bipolar transistors based on GaAlAs/GaAs and InAlAs/InGaAs materials. In 1991, Dr. Asbeck joined the University of California at San Diego, where he is the Skyworks Chair Professor in the Department of Electrical and Computer Engineering. His research interests are in development of high performance transistor technologies and their circuit applications. Dr. Asbeck's research has led to more than 300 publications. He is a member of the NAE, and has been a Distinguished Lecturer of the IEEE Electron Device Society and of the Microwave Theory and Techniques Society. He received the 2003 IEEE David Sarnoff Award for his work on heterojunction bipolar transistors.



Prabhakar R. Bandaru received his Ph.D. from U.C. Berkeley in 1998, in the area of magneto-optic memories. He then worked at Applied Materials Inc. on the development of ferroelectric non-volatile memories. Subsequently, as a postdoctoral fellow in the Electrical Engineering department at UCLA, he conducted research in solid state quantum computing and communications. In 2003, Dr. Bandaru joined the University of California at San Diego, where he is an Associate Professor of Materials Science in the Department of Mechanical Engineering and an affiliate Professor in the Department of Electrical and Computer Engineering. Dr. Bandaru's research accomplishments span the areas of carbon nanotubes, semiconductors, optics, and magnetics. He has worked extensively on nonlinear nanotube structures, such as Y-junctions and helical morphologies, for electronic device applications. His current research interests include the development of novel nanoelectronics using nanotubes and nanowires, thermoelectric materials for energy conversion, nanocomposites, techniques for nano-patterning, nanophotonics, and single electron and single photon devices. Dr. Bandaru's research has led to more than 50 publications. He received the 2007 NSF CAREER Award and was named to the Scientific American 50, in 2006, for his contributions to carbon nanotube electronics.