

CARBON NANOTUBE/CONDUCTIVE ADDITIVE/SPACE DURABLE POLYMER NANOCOMPOSITE FILMS FOR ELECTROSTATIC CHARGE DISSIPATION

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ABSTRACT

Thin film membranes of space environmentally stable polymeric materials possessing low color/solar absorptivity (α) are of interest for potential applications on Gossamer spacecraft. In addition to these properties, sufficient electrical conductivity is required in order to dissipate electrostatic charge (ESC) build-up brought about by the charged orbital environment. One approach to achieve sufficient electrical conductivity for ESC mitigation is the incorporation of single wall carbon nanotubes (SWNTs). However, when the SWNTs are dispersed throughout the polymer matrix, the nanocomposite films tend to be significantly darker than the pristine material resulting in a higher α . The incorporation of conductive additives in combination with a decreased loading level of SWNTs is one approach for improving α while retaining conductivity. Taken individually, the low loading level of conductive additives and SWNTs is insufficient in achieving the percolation level necessary for electrical conductivity. When added simultaneously to the film, conductivity is achieved through a synergistic effect. The chemistry, physical, and mechanical properties of the nanocomposite films will be presented.

KEY WORDS: Low Color Polyimides, Polyimides, Nanocomposites, Electrostatic Charge Mitigation, Carbon Nanotubes, Space Durable Polyimides

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1. INTRODUCTION

Several space mission concepts proposed by NASA are based on the use of large, deployable, and ultra-lightweight vehicles (e.g. Gossamer spacecraft) (1). These spacecraft consist of both structural and polymer film components. One example of a 'proof of concept' of this approach was the Inflatable Antennae Experiment (IAE) deployed from the space shuttle Endeavor (STS-77) in 1996 (2). The experiment involved the successful deployment of a 14 meter Mylar® film based antennae from a compact vehicle (Spartan satellite). Since this experiment was a proof of concept, the space environmental durability of the materials was not of concern.

Future Gossamer spacecraft will require films that are durable to the space environment and compliant. Compliance is needed so that the film can be folded into the compact volumes that are suitable for conventional launch vehicles. Once in orbit, the folded films are deployed to create structures that are many square meters in size. To be space durable, the film must exhibit a unique combination of physical and mechanical properties. These properties are orbit dependent and include resistance to many environmental factors such as atomic oxygen (AO) in LEO, ultraviolet (UV) and vacuum UV radiation, and electron and proton attack. For some applications the film must also exhibit low solar absorptivity (α) and high thermal emissivity (ϵ). In addition to these properties, the film must exhibit sufficient electrical conductivity to mitigate electrostatic charge (ESC) build-up without degrading other desired properties (e.g. flexibility, α , ϵ , transparency). Since polymeric films are inherently insulative, they can become charged and behave like a capacitor. Discharge can happen in a single event causing considerable damage to surrounding materials and electronics on the vehicle. To mitigate ESC build-up, a surface resistance in the range of 10^6 to 10^{10} ohms/square is needed.

Many of these material requirements can be met by aromatic polyimides, due to their excellent physical and mechanical properties as well as radiation resistance. Through the proper choice of monomers, low color (which is related to α) and AO resistance have been achieved (3,4). Electrical conductivity necessitates the incorporation of uniformly dispersed additives in the polymer. One such additive that has received much attention is single walled carbon nanotubes (SWNTs) (5-11). Based on their inherent size and electrical properties, SWNTs are excellent candidates for obtaining the surface resistance necessary for ESC dissipation. However, good and uniform dispersal of SWNTs has been challenging to achieve due their insolubility and/or incompatibility with the host resin. In general, SWNTs tend to agglomerate as bundles in solution and if dispersed, reaggregate soon thereafter due to electrostatic attraction.

Uniform dispersions of SWNTs have been achieved in space durable polymers (e.g. LaRC™ CP2) by an in-situ polymerization approach (5,7,8) and by the addition of the SWNTs into amide acid polymers terminated with alkoxy silane moieties (7,11). By both approaches, bulk conductivity sufficient for ESC mitigation was achieved at a weight loading of 0.05% (w/w)

relative to the polymer. There was, however, a loss in the optical transparency of the film and an increase in α compared to the virgin material. Other approaches have involved the preparation of surface coatings (9,10) that only give surface, not volume conductivity.

One method of improving the optical and thermo-optical properties while retaining sufficient electrical conductivity would involve the introduction of a low amount of a second conducting agent with simultaneous reduction of the quantity of SWNTs. Ideally, this second conductive additive would be anticipated to have a negligible effect upon these properties. It has been reported that electrical conductivity was achieved at a volume loading of 0.06% conductive carbon black in an epoxy by the addition of a small amount of an inorganic salt (12). This was proposed to be due to the agglomeration of the carbon black into networks throughout the matrix by increasing the ionic concentration (strength) of the epoxy. Thus it was postulated that by increasing the ionic strength of the polyimide matrix by the addition of an inorganic salt (second conducting agent), sufficient network formation of SWNTs at a low loading level could be achieved. This in turn would presumably improve the optical and thermo-optical properties of the film due to decreased loading levels of SWNTs. The preparation and characterization of nanocomposite films based on the addition of the SWNTs into amide acid polymers terminated with alkoxy silane moieties approach is described herein.

2. EXPERIMENTAL

2.1 Starting Materials Aminophenyltrimethoxysilane (APTS, Gelest Inc., ~90% meta, ~10% para) was purified by vacuum distillation. 4,4'-Hexafluoroisopropylidene diphthalic anhydride [6FDA, Hoechst Celanese Inc., melting point (m.p.) 241-243 °C] was sublimed prior to use. 1,3-Bis(3-aminophenoxy)benzene (APB, Mitsui Chemicals America, Inc. m.p. 107-108.5 °C) and N,N-dimethylacetamide (DMAc) were used as received without further purification. HiPCO SWNTs were obtained from Tubes@Rice. The SWNTs were purified by soxhlet extraction with hydrochloric acid for ~24 hours, then treated at 250°C for 8 hours in a high humidity chamber. All other chemicals were obtained from commercial sources and used as received without further purification.

2.2 Polymer Synthesis A typical procedure for the preparation of the alkoxy silane terminated amide acid (ASTAAs) is described. Into a 500 mL three neck round-bottom flask equipped with a mechanical stirrer, nitrogen gas inlet, and drying tube filled with calcium sulfate were placed APB (22.8551 g, 0.0782 mol), APTS (0.8552 g, 0.0040 mol), and DMAc (50 mL). Once dissolved, the flask was immersed in a water bath to regulate the temperature. 6FDA (35.6215 g, 0.0802 mol) was added in one portion as a slurry in DMAc (45 mL) and rinsed in with 25 mL of DMAc to afford a solution with a solids content of ~34.4% (w/w). The reaction was stirred for ~24 hours at ambient temperature under nitrogen. The inherent viscosity (η_{inh}) in DMAc at 25°C for a 0.5% (w/v) solution was 0.57 dL/g.

2.3 Blending of SWNT and ASTAA The following example is representative of the preparative method employed. To a 100 mL round bottom flask equipped with nitrogen inlet, mechanical stirrer, and drying tube filled with calcium sulfate was charged 11.06 g of an ASTAA solution (~33.5% solids). In a separate tared vial, SWNTs (0.0011 g) were placed in 3 mL of DMAc and the mixture sonicated in a water bath at room temperature for ~2.5 hours. The suspended tubes were added to the stirred mixture of ASTAA at room temperature and rinsed in with 4.5 mL of DMAc to afford a solids content of ~20%. The SWNT concentration was 0.03% by weight relative to the ASTAA. The mixture was stirred for ~16 hours under a nitrogen atmosphere at room temperature prior to film casting.

2.4 Blending of salt and ASTAA The following example is representative of the preparative method employed. To a 100 mL round bottom flask equipped with nitrogen inlet, mechanical stirrer, and drying tube filled with calcium sulfate was charged 5.8 g of an ASTAA solution (~33.7% solids). Copper sulfate (0.0003 g) was added and rinsed in with 4 mL of DMAc to afford a solids content of ~20%. The salt concentration was 0.014 % by weight relative to the ASTAA. The mixture was stirred for ~16 hours under a nitrogen atmosphere at room temperature prior to film casting.

2.5 Blending of SWNT, salt, and ASTAA The following example is representative of the preparative method employed. To a 100 mL round bottom flask equipped with nitrogen inlet, mechanical stirrer, and drying tube filled with calcium sulfate was charged 9.82 g of an ASTAA solution (~34.4% solids). In a separate tared vial, SWNTs (0.0010 g) and copper sulfate (0.0007 g) were placed in 3 mL of DMAc and the mixture sonicated in a water bath at room temperature for ~2.5 hours. The suspended tubes and salt mixture were added to the stirred ASTAA solution at room temperature and rinsed in with 4 mL of DMAc to afford a solids content of ~20%. The SWNT concentration was 0.03% and the salt concentration was 0.014%, respectively, by weight relative to the ASTAA. The mixture was stirred for ~16 hours under a nitrogen atmosphere at room temperature prior to film casting.

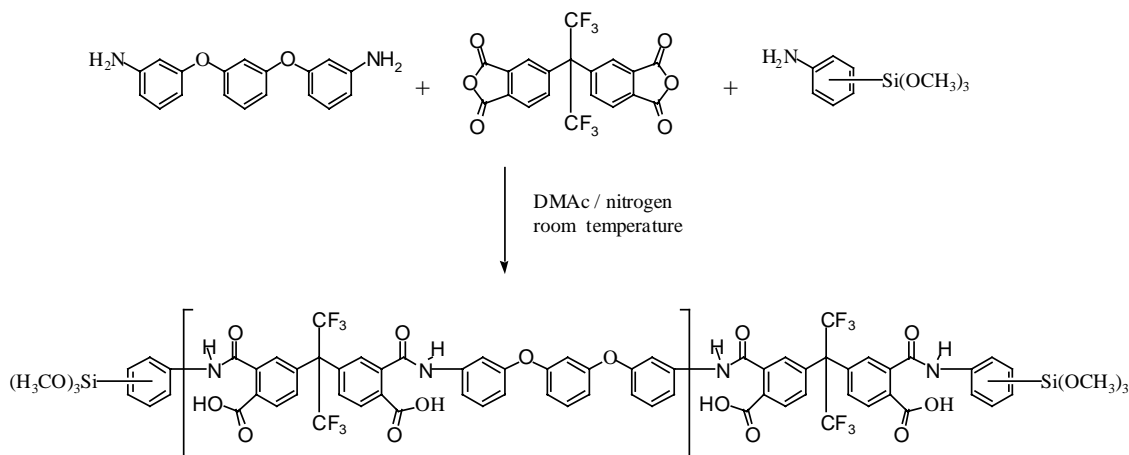
2.6 Films DMAc solutions of the neat ASTAA and nanoparticle containing ASTAAs were doctored onto clean, dry plate glass and dried to tack-free state in a low humidity chamber at ambient temperature. The films were cured in a forced air oven at 100, 200, and 300 °C for 1 hour each. The films were subsequently removed from the glass plate and characterized.

2.7 Other Characterization Melting point ranges (tangent of onset to melt and the endothermic peak) were determined by either differential scanning calorimetry (DSC) at a heating rate of 10 °C/min or visually on a Thomas-Hoover capillary melting point apparatus (uncorrected). Glass transition temperatures (T_g s) were determined on thin films at a heating rate of 20 °C/min and were taken as the inflection point of the ΔH vs. temperature curve. Optical

microscopy was performed on an Olympus BH-2 at a magnification of 200X. The % transmission at 500 nm was obtained on thin films using a Perkin-Elmer Lambda 900 UV/VIS/NIR spectrometer. Solar absorptivity (α) was measured on an AZ Technology Model LPSR-300 spectrophotometer with measurements taken between 250 to 2800 nm using a vapor deposited aluminum on Kapton® film (1st surface mirror) as a reflective reference for air mass 0 per ASTM E903. An AZ Technology Temp 2000A infrared reflectometer was used to measure thermal emissivity (ϵ). Surface resistivity was measured using a Prostat® PSI-870 Surface Resistance and Resistivity Indicator per ASTM D-257 and was reported as an average of three readings. Volume conductivity was measured using a Prostat® PSI-801 Resistance System with a PRF-911 Concentric Ring Fixture per ASTM D-257.

3. RESULTS AND DISCUSSION

3.1 Polymer Synthesis In this study, the addition of nanoparticles to a premade amide acid polymer terminated with alkoxy silane (ASTAA) groups approach was followed (7,11). The ASTAA was based on the LaRC™ CP2 backbone. It was prepared by the reaction of 6FDA with APB and end-capped with APTS at a 2.5% molar offset (Scheme 1) corresponding to a calculated number average molecular weight (\bar{M}_n) of ~27,700 g/mole. To mitigate the temperature increase of the solution when 6FDA was added, the flask was immersed in a water bath. This was done to decrease the likelihood for premature reaction of the alkoxy silane groups. It was then allowed to stir overnight under a nitrogen atmosphere at ambient conditions. The polymer was prepared several times with η_{inh} s ranging from 0.57 to 0.78 dL/g.



Scheme 1. Alkoxy silane-Terminated Amide Acid of LaRC™ CP2 Synthesis

3.2 Blending of Nanoadditives with LaRC™ CP2 ASTAA The nanoadditives evaluated were HiPCO SWNTs, inorganic salts, and a combination of the inorganic salt with the SWNTs. Previous analysis of the SWNTs by atomic force microscopy indicated that the tubes were roped into large bundles ~25 nm in diameter. The diameter of the individual tubes could not be discerned due to the large packing density. Additionally, the length scales of the individual tubes could not be determined due to the tubes bundling into ropes. However, these appeared to be typical SWNTs consistent with other HiPCO prepared materials with the following dimensions: ~0.7 to 1.5 nm in diameter and in the micron range for length (7). The SWNTs were incorporated at loadings of 0.03 and 0.04 weight %. Prior to addition to the ASTAA solution, the SWNTs in DMAc were sonicated for ~2.5 hours to afford a SWNT suspension.

The inorganic salts investigated were copper sulfate (CuSO_4), copper chloride (CuCl_2), and cobalt chloride (CoCl_2). These salts were incorporated at loadings of 0.014 and 0.042 weight %. The loading level of 0.014 % corresponded to the value of 3×10^{-6} mol that was determined to be the quantity needed to achieve conductivity in an epoxy matrix containing conductive carbon black (12). The CuCl_2 was chosen based on the work performed with the epoxy. The other two salts were chosen based on their relative absorptive properties in the region of interest (500 nm). Since the salts were soluble in DMAc, they were added directly to the stirred ASTAA solution.

Blends of the inorganic salts with the SWNTs were evaluated at two different weight loadings of SWNTs. Prior work indicated that the 0.03% loading was non-conductive while a 0.05% loading was conductive. In this work, a 0.04% loading level was evaluated. The salts mixed with the SWNTs were CuSO_4 and CoCl_2 . The CuCl_2 was not chosen for this part of the work based on the effect on the % transmittance as described in section 3.3. The same procedure was followed as described above for the SWNTs but with the addition of the salt.

In all cases, the solutions were stirred overnight under a nitrogen atmosphere at ambient conditions prior to film casting. Once dried to a tack free state, the films were cured to 300°C in flowing air to effect imidization and condensation of the silanol endgroups. Due to the acidic nature of the amide acid, no additional acid catalyst was required for the hydrolysis and condensation of the alkoxy silane endgroups. All of the cured films exhibited a T_g of ~206°C.

3.3 Optical Transparency Optical transparency at 500 nm, the solar maximum, was determined on thin films with the results presented in Table 1. Since thickness has an effect upon the optical properties, care was taken to obtain films that were of comparable thickness. The thickness ranged from 45 to 48 μm ; except for one (P2) that was 55 μm . The addition of the various salts at a 0.014% (w/w) loading was found to decrease the % transmission compared to the base polymer (P1) in the order of P4 (78%) > P2 (73%) > P3 (69%). The cured copper based films exhibited an orange-brown color, while the cured cobalt based film had a blue hue.

Table 1. Optical Transparency at 500 nm

Film ID	SWNT, % (w/w)	Additive, %(w/w)	Film Thickness, μm	Transmission @ 500 nm, %
P1	---	---	48	85
P2	---	CuSO ₄ , 0.014	55	73
P3	---	CuCl ₂ , 0.014	48	69
P4	---	CoCl ₂ , 0.014	48	78
P5	0.03	----	45	66
P6	0.03	CuSO ₄ , 0.014	48	63
P7	0.03	CuSO ₄ , 0.042	45	53
P8	0.04	-----	48	58
P9	0.04	CuSO ₄ , 0.014	45	59

Due to the lower % transmission value exhibited by the CuCl₂ containing film (P3), no further work was performed. As observed in earlier studies, increasing SWNT concentration led to a darkening of the film with P1 > P5 > P8 (5,7,8,11). When CuSO₄ was added to the 0.03% (w/w) SWNT composition, the % transmission decreased with increasing salt concentration in the order of P5 > P6 > P7. Comparable % transmissions were obtained for films P8 and P9 containing 0.04% SWNTs even though P9 contained 0.014% CuSO₄ and maybe due to slight differences in thickness.

3.4 Optical Microscopy To assess the effect of salt addition upon the 0.03 and 0.04% weight loadings of SWNTs, P5-P9 were examined by optical microscopy at 200X (Fig. 1). The pristine polymer (P1) is featureless and is shown as a comparison. Films P5 and P6, containing only SWNTs, and P6, containing 0.014% (w/w) CuSO₄, exhibited good and uniform dispersion of SWNTs. Film P5 containing 0.03% (w/w) SWNT exhibited a “peppered” appearance of SWNTs with no apparent connectivity. With the addition of 0.014% (w/w) CuSO₄ (P6), connectivity or network formation of the SWNTs was observed. The micrograph of P6 shows a “matted” appearance of the SWNTs. A similar effect was reported for the addition of salt to conductive carbon black in an epoxy (12). Increasing the salt concentration to 0.042% (P7) resulted in enhancing this effect. For P8 containing 0.04% (w/w) SWNTs, this network or “matted” appearance was evident. The addition of CuSO₄ (P9) resulted in enhancing this network and was comparable in appearance to that of P7. For films P7 and P9, the SWNTs appeared uniformly dispersed but tended to be more bundled.

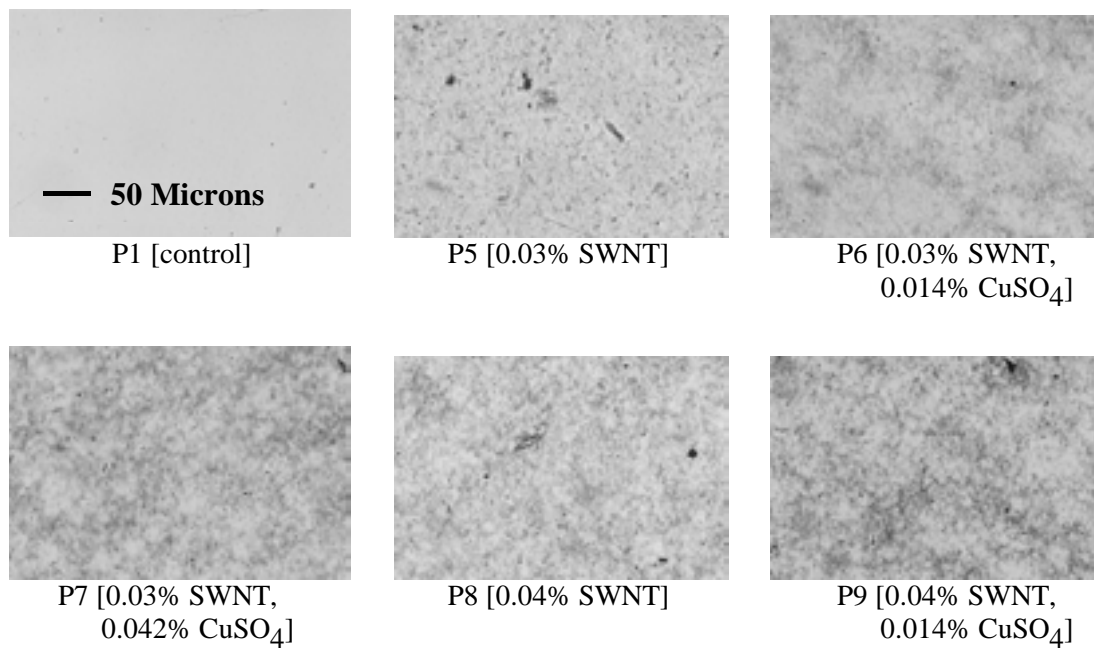


Figure 1: Optical Micrographs at 200X of Films P1 and P5 – P9.

3.5 Thermo-optical Properties of Nanocomposites The effect of salt inclusion in the SWNT containing films on the solar absorptivity (α) and thermal emissivity (ϵ) was of interest since these properties are important for some mission applications. The solar absorptivity (α) pertains to the fraction of incoming solar energy that is absorbed by the film and is typically low (~ 0.1) for a low color film. The thermal emissivity (ϵ) is a measure of the films ability to radiate energy from the film surface. The results are tabulated in Table 2. In general, the addition of the copper salts at 0.014% (w/w) increased α by $\sim 30\%$ with minimal effect on ϵ as compared to P1. Addition of CoCl₂ at this loading level had no effect upon the thermo-optical properties as compared to P1. As previously reported, increasing % SWNT loading increased α and ϵ in the order: P8 > P5 > P1 (5,7,8,11). When CuSO₄ was added to the 0.03% (w/w) SWNT composition, α increased with increasing salt concentration with P7 > P6 > P5. The ϵ was essentially the same regardless of salt concentration. At a 0.04% (w/w) loading of SWNTs (P8 and P9), the inclusion of salt had no effect upon α and ϵ .

Table 2. Thermo-optical Properties

Film ID	SWNT, % (w/w)	Additive, % (w/w)	Solar Absorptivity, α	Thermal Emissivity, ϵ
P1	---	---	0.09	0.58
P2	---	CuSO ₄ , 0.014	0.12	0.60
P3	---	CuCl ₂ , 0.014	0.12	0.58
P4	---	CoCl ₂ , 0.014	0.08	0.58
P5	0.03	----	0.22	0.61
P6	0.03	CuSO ₄ , 0.014	0.28	0.63
P7	0.03	CuSO ₄ , 0.042	0.31	0.64
P8	0.04	-----	0.30	0.65
P9	0.04	CuSO ₄ , 0.014	0.31	0.64

3.6 Surface and Volume Resistivity Electrical conductivity was determined under ambient conditions with the results presented in Table 3. As expected, the pristine material (P1) and films P2 – P4 were insulative. Film P5 containing 0.03 % (w/w) SWNT was insulative, while P8 containing 0.04% (w/w) SWNTs exhibited a surface resistivity of $10^8 \Omega/\text{square}$ and a volume conductivity of $10^{10} \Omega \text{ cm}$. This level of conductivity is sufficient for ESC mitigation as previously mentioned. These results are supported by the optical micrographs (section 3.4) where P5 exhibited a “peppered” appearance of the SWNTs with no connectivity whereas P8 exhibited a “matted” appearance suggestive of a network. Prior work suggested the percolation threshold necessary for conductivity was between 0.03 and 0.05% SWNT loading (7). Theoretical predictions have placed the percolation level needed for attaining conductivity at 0.05 volume % (8). In another report, experimental data using untreated catalytically grown SWNTs in an epoxy matrix suggested that the electrical percolation threshold was between 0.0225 and 0.04% (w/w) (13).

The addition of 0.014% (w/w) CuSO₄ to a mix containing 0.03% (w/w) SWNTs resulted in a film exhibiting a surface resistivity of $10^8 \Omega/\text{square}$ and a volume conductivity of $10^{10} \Omega \text{ cm}$. These values were comparable to that of P8 that contained 0.04% (w/w) SWNT and no salt. The addition of the salt resulted in increasing the ionic strength of the matrix (12). This presumably caused the SWNT to agglomerate and form a sufficient network to provide conductivity. The optical micrograph of P6 in section 3.4 tends to support this network formation. Increasing the salt concentration (P7 compared to P6) afforded one order of magnitude improvement in surface resistivity. The volume resistivity though remained unchanged. When 0.014% (w/w)

Table 3. Surface & Volume Resistivity Characterization

Film ID	SWNT, % (w/w)	Additive, %(w/w)	Surface Resistivity, Ω /square	Volume Resistivity, Ω cm
P1	---	---	$>10^{12}$	8.5×10^{15}
P2	---	CuSO ₄ , 0.014	$>10^{12}$	8.7×10^{15}
P3	---	CuCl ₂ , 0.014	$>10^{12}$	9.1×10^{15}
P4	---	CoCl ₂ , 0.014	$>10^{12}$	4.7×10^{15}
P5	0.03	----	$>10^{12}$	9.9×10^{14}
P6	0.03	CuSO ₄ , 0.014	10^8	1.9×10^{10}
P7	0.03	CuSO ₄ , 0.042	10^7	1.5×10^{10}
P8	0.04	-----	10^8	1.7×10^{10}
P9	0.04	CuSO ₄ , 0.014	10^8	2.0×10^{10}

CuSO₄ was added to a SWNT composition (P9) that already displayed conductivity (P8), the surface and volume resistivity remained unchanged. However, there was more apparent SWNT agglomeration in P9 as compared to P8 as discussed in section 3.4.

4. SUMMARY

Films with surface and volume resistivities sufficient to mitigate ESC build-up (10^6 to 10^{10} ohms/square) were prepared by the incorporation of a low loading level of SWNTs in conjunction with a small amount of salt. The salt was proposed to increase the ionic strength of the matrix thereby resulting in sufficient network formation of the SWNTs to afford conductivity. Optical micrographs suggested that this occurred. Addition of salt to a SWNT containing film that was conductive resulted in increased agglomeration of the tubes with no increase in surface resistivity. At a 0.03% (w/w) SWNT loading, the CuSO₄ was found to increase α and ϵ , but not as much as a 0.04% (w/w) SWNT loading.

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John W. Connell is a senior polymer scientist in the AMPB of the SMC at NASA LaRC. He received B.S. and Ph. D. degrees from Virginia Commonwealth University in 1982 and 1986, respectively. Prior to joining NASA LaRC in January 1988, he was a research associate at Virginia Commonwealth University. Since coming to NASA, his work has focused on the development of high performance polymers for aerospace applications.