

Transparent, flexible, conductive carbon nanotube coatings for electrostatic charge mitigation[☆]

Kent A. Watson^{a,*}, Sayata Ghose^{b,1}, Donavon M. Delozier^{b,1},
Joseph G. Smith Jr^b, John W. Connell^b

^aNational Institute of Aerospace, 144 Research Drive, Hampton VA 23666-1399, USA

^bNational Aeronautics and Space Administration, Langley Research Center, MS 226, Hampton VA 23681-2199, USA

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Abstract

Low color, space environmentally durable polymeric films with sufficient electrical conductivity to mitigate electrostatic charge (ESC) build-up are needed for applications on advanced spacecraft, particularly on large, ultra-light weight space structures. For these applications, the films must exhibit electrical conductivity that can survive the folding and unfolding required for packaging and deployment. The work described herein consists of coating the surface of polymer films with a thin layer of single-walled carbon nanotubes (SWNTs). Surface resistivities in the range sufficient for ESC mitigation were achieved with minimal effects on the optical and thermo-optical properties of the films. Bending, folding or crumpling did not affect the surface resistivity of the coated films. The preparation and characterization of SWNT coated space durable polymer films are discussed.

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1. Introduction

Large, deployable, ultra-lightweight spacecraft require polymeric materials (e.g. films) that possess a unique combination of mechanical and optical properties [1]. The mission and orbital environment of the spacecraft determine specific performance requirements for the materials. Some of the desired properties include atomic oxygen (AO) resistance [for spacecraft in low Earth orbit (LEO)], low solar absorptivity (low color), high thermal emissivity, resistance to ultraviolet (UV) and vacuum UV radiation, good mechanical properties, good processability, tear resistance, the ability to be folded and unfolded without loss of desired properties, and sufficient electrical

conductivity for dissipation of electrostatic charge (ESC). The goal of this work was to prepare transparent, flexible, anti-static, space durable polymer films to satisfy material needs for future NASA missions.

Many of the material requirements for space applications are met by using aromatic polyimides due to their exceptional mechanical properties as well as resistance to UV and charged particle radiation. By proper choice of the monomers, low color (low solar absorptivity) and AO resistant polyimides have been reported [2,3]. However, the incorporation of sufficient electrical conductivity into polymer films to mitigate ESC build-up without adversely effecting the solar absorptivity (α) and flexibility has been difficult to attain.

Particles prevalent in the Earth's orbital environment include electrons/ions, solar flare protons and galactic cosmic rays [4–6]. The high energies of these particles allow them to penetrate spacecraft surface layers and cause chemical reactions that can ultimately degrade optical and/or mechanical properties of organic polymers. Furthermore, energetic charged particles—particularly electrons—can

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* Corresponding author. Tel.: +1 757 864 4287; fax: +1 757 864 8312.

E-mail address: k.a.watson@larc.nasa.gov (K.A. Watson).

¹ National Research Council Research Associate located at NASA Langley Research Center.

penetrate surface layers, depositing a charge onto insulating materials and other electrically isolated surfaces on a spacecraft. This can result in ESC build-up and development of large electric fields. Consequent discharging can potentially cause catastrophic damage to sensitive spacecraft electronics [7,8]. ESC build-up is also a problem during the manufacturing of polymer films, particularly ultra-thin films. As a result, handling and manipulation of the film during subsequent fabrication processes can be problematic. The surface resistivity (which relates to surface conductivity) required to dissipate ESC build-up on insulators (such as polymer films) is in the range from 10^6 to 10^{10} Ω /square.

One approach to lowering the surface resistivity on polymer films without severely detracting from the desired optical and thermo-optical properties is through the use of single-walled carbon nanotubes (SWNTs). Discovered in the early 1990s [9,10], SWNTs exhibit great potential for improving the electrical properties of materials. However, achieving good dispersion of SWNTs throughout a polymer matrix has been difficult due to the insolubility of the SWNTs and/or incompatibility with the host resin. SWNTs tend to agglomerate in solvents and the host resin. Even if initially dispersed, they may re-agglomerate soon thereafter depending upon the viscosity of the matrix.

Reasonable dispersions of SWNTs in some space durable polymers have been reported [11–15]. This was achieved by using both an in situ polymerization approach and the addition of SWNTs to amide acid polymers containing reactive end groups [15]. The first method involved the preparation of the amide acid in the presence of the SWNTs with simultaneous ultrasonic treatment followed by thermal conversion to the polyimide. Alternatively, the amide acid/SWNT mixture can be cyclodehydrated chemically in solution provided that the polyimide remains soluble. The second approach involved the dispersion of SWNTs in solutions of alkoxysilane terminated amide acid oligomers. In both of these approaches, the SWNTs were dispersed throughout the polymer matrix and consequently the nanocomposite films exhibited similar surface and volume resistivities. When the SWNT loading level was ≥ 0.05 wt%, surface and volume resistivities sufficient for ESC mitigation were achieved. However, in these films there was notable loss of the optical transparency and an increase in the solar absorptivity compared to the host polymer.

Recently, work on adding a combination of inorganic salts and SWNTs to polymers was reported [16]. Films with surface and volume resistivities sufficient to mitigate ESC build-up were prepared by the incorporation of a low loading level of SWNTs in conjunction with a small amount of inorganic salt. Salt inclusion to a 0.03 wt% SWNT loading afforded materials with comparable resistivity values to that of a 0.04 wt% SWNT loading; however, the optical and thermo-optical properties were not significantly improved.

Many applications for polymers on spacecraft require only surface resistivity to mitigate ESC (volume conductivity is not necessary) [17,18]. The amount of SWNTs necessary to mitigate ESC build-up is greatly reduced when only surface resistivity is required as compared to films containing SWNTs dispersed throughout the bulk. The use of a lower amount of SWNTs would be expected to have negligible effects on the optical and thermo-optical properties of the films. The preparation and characterization of space durable polyimide films surface coated with SWNTs is described herein. Spray-coating techniques were used to apply the SWNTs to three space durable polymers. Films coated with SWNTs were compared with uncoated films as well as those prepared via in situ polymerization.

2. Experimental

2.1. Starting materials

1,3-Bis(3-aminophenoxy)benzene (APB, Mitsui Chemicals America, Inc., mp 107–108.5 °C) was used without further purification. 4,4'-Oxydiphthalic dianhydride (ODPA, Imitec, Inc., mp 224–225.5 °C) and 4,4'-hexafluoroisopropylidene diphthalic anhydride (6FDA, Hoechst Celanese Inc., mp 241–243 °C) were sublimed prior to use. [2,4-Bis(3-aminophenoxy)phenyl]-diphenylphosphine oxide (3-APPO, mp 195–196.5 °C) was prepared as previously reported [2]. HiPco SWNTs (BuckyPearls™) were obtained from Carbon Nanotechnologies Inc. (batch # P0222, purified) and treated as described below. *m*-Cresol (Fluka Chemical Co.) was vacuum distilled prior to use. Isoquinoline, *N,N*-dimethylacetamide (DMAc, anhydrous), *N*-methyl-2-pyrrolidinone (NMP, anhydrous), tetrahydrofuran (THF, anhydrous), 1,2-dichlorobenzene (DCB), sodium methoxide (powder), pyridine, and acetic anhydride were used as received (Aldrich Chemical Co.) without further purification.

2.2. SWNT treatment

SWNTs were treated as follows: a 250 ml round-bottom flask was charged with SWNTs (0.100 g) and DCB (220 ml). The mixture was sonicated for 3 h at room temperature. In a separate flask, pyridine (80 ml) and sodium methoxide (0.264 g) were stirred under nitrogen at room temperature. The SWNT/DCB mixture was added to the flask containing the pyridine and sodium methoxide and the resultant mixture heated to 80 °C. After 16 h at 80 °C, the temperature of the reaction mixture was increased to ~ 115 °C and the pyridine was removed by distillation. The reaction mixture was then stirred in refluxing DCB for 3 h followed by cooling to room temperature. The SWNTs were isolated via centrifugation, washed several times with methanol, and dried in a vacuum oven at 150 °C for 6 h. The treated SWNTs formed a stable suspension in DMAc, DMF,

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