Single-wall carbon nanotube coating on a pyroelectric detector

John H. Lehman, Chaiwat Engtrakul, Thomas Gennett, and Anne C. Dillon

Carbon single-wall nanotubes (SWNTs) are studied as the thermal-absorption coating on a large area pyroelectric detector. The SWNTs were produced by a laser vaporization method and dispersed onto the detector surface by use of a simple airbrush technique. The detector was based on a 1-cm-diameter, 60-μm-thick lithium tantalate disk having nickel electrodes. We report the spectral responsivity of the detector ranging from 600 to 1800 nm, as well as the spatial and directional uniformity at 850 nm. Using Drude and Lorentzian dielectric functions and an effective medium approximation to obtain the indices of refraction of semiconductor and metallic SWNTs, we compared the expected theoretical relative responsivity for the two types of tube with the measured relative responsivity of the detector. Values of thermal conductivity, specific heat, and damage threshold obtained from the literature are compared with properties of alternatives for thermal coatings such as gold-black and carbon-based paint.

OCIS codes: 040.0040, 040.5150.

1. Introduction

Recently, several groups have undertaken tasks to evaluate the optical properties and to generate theoretical models to predict the material and electronic properties of carbon nanotubes.[1–3] We report on the process of applying carbon single-wall nanotubes (SWNTs) to a pyroelectric detector and further explore the optical and thermal properties of SWNTs. To the best of our knowledge, the pyroelectric detector coated with SWNTs is the first of its kind.

When designing any thermal detector (bolometer, thermopile, etc.), we normally want to achieve both the highest sensitivity and the lowest noise equivalent power uniformly over a wide wavelength range. The spectral and thermal properties of the coating and detector combination must be optimized within the constraints of the composite thermal conductivity, specific heat, and mass. In principle, the spectral responsivity and spatial uniformity of a pyroelectric detector (or any thermal detector) depends on the absorption of the detector coating as a function of wavelength. Pyroelectric detectors are capable of broad and uniform spectral responsivity from the visible to the near infrared as well as relative variations in spatial and directional uniformity that are less than 1%.[4] During the past 50–100 years, thermal coatings have been developed from carbon-based paints, diffuse metals (for example, gold black), and oxidized metals.[5] Gold-black coatings are capable of low reflectance over the wavelength range from 0.2 to beyond 50 μm. However, such coatings can contribute substantially to the thermal mass of the detector and are vulnerable to damage from heating, aging, hardening, and physical contact.[6]

Carbon nanotubes are known to be lightweight inert materials with high thermal conductivities.[7] Experimental evidence with regard to other thermal properties such as specific heat and damage resistance indicates that carbon-nanotube coatings can be superior to present alternatives.[8] We have undertaken the evaluation of a SWNT-coated pyroelectric detector by comparing its spectral responsivity with a state-of-the-art pyroelectric detector coated with gold black. Measurements of the spatial and directional uniformity as well as general comments on the thermal properties are reported.

2. Detector Fabrication and Coating

Two pyroelectric detectors were fabricated. They were identical except that one was coated with gold black (the gold-black-coated detector) and the other with SWNTs (the SWNT-coated detector). A photograph of

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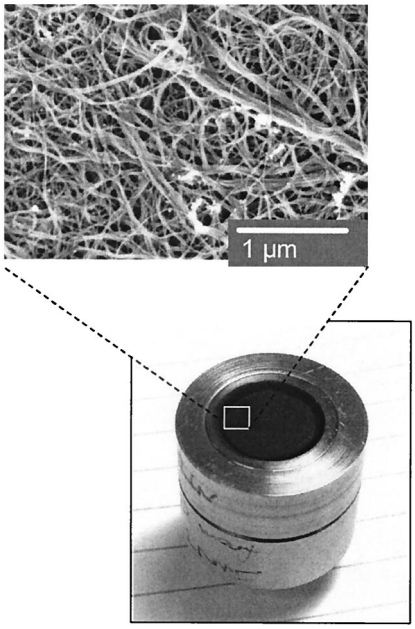

Fig. 1. Pyroelectric detector coated with SWNTs (detector 2). The active detector area is 1 cm in diameter. Inset, appearance of the tubes imaged by a scanning electron microscope (1-μm scale as indicated).

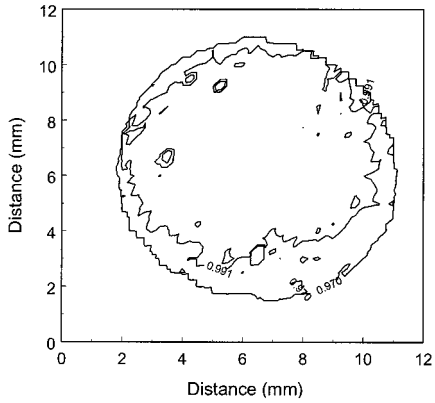

Fig. 2. SWNT-coated detector response as a function of position at 850-nm wavelength (1% contour intervals).

the SWNT-coated detector is shown in Fig. 1. Each detector was constructed from a 12-mm-diameter, 60-μm-thick lithium tantalate ($LiTaO_3$) disk, with 10-mm-diameter and 25-nm-thick nickel (Ni) electrodes on each face. The gold-black coating was deposited in a 2.6-Torr nitrogen environment as described in previous work.[4,6] The synthesis of the SWNTs was accomplished by use of a laser vaporization method similar to that of Guo *et al.*,[9] whereby an alexandrite laser operating at a wavelength of 755 nm and a power density of 40 W/cm^2 was employed to vaporize a graphite target doped with cobalt (0.6% relative atomic mass) and Ni (0.6% relative atomic mass). The crude soot was produced at 1200 °C with 500-Torr Ar flowing at 100 cm^3/min (units based on standard air). The SWNTs were purified with a HNO_3 reflux for 16 h, followed by air oxidation at 550 °C for 30 min as described in an earlier publication.[10] The purified SWNTs were dispersed in chloroform by use of a cup-horn sonicator connected to an ultrasonic processor. During the entire sonication period (2 h), the suspensions were kept in a water bath cooled to 15 °C. The SWNT (0.1 wt. %) suspension was then applied from 3×2 aliquots by use of an airbrush and dried under a steady stream of nitrogen.

The exact thickness of the SWNT coating was estimated with a scanning electron microscope to be between 0.5 and 2 μm, with images from similar depositions on glass slides. For modeling purposes, the SWNT coating thickness was determined to be 2 μm thick.

With the availability of purified nanotubes in bulk form, the prospect of using an airbrush to apply the SWNT coating is attractive compared with depositing gold black. Unlike the gold-black deposition process, the coating can be applied after the detector is completely assembled and risk of damage to the detector is minimal. After drying, the SWNT coating is not easily dislodged by forced air. Therefore, unlike gold-black-coated detectors, dust can be removed from the detector surface without damaging the coating.

3. Experimental Results

The spatial and directional uniformity of the SWNT-coated detector was measured with a diode laser mounted on an industrial robot arm. The diode laser operated at a wavelength of 850 nm, was actively stabilized to prevent thermal drift and output power variations, and had an output beam size of approximately 1 mm (that is, greater than 99% of the circular beam was contained within a 1-mm diameter). The laser light was modulated and the detector output was sampled by means of a lock-in amplifier and thereby relative-detector response variations were measured independent of background light. The uncertainty of the spatial and directional uniformity measurements has not been fully evaluated, but we currently assign a value of 0.5% (type B).

We measured the detector spatial uniformity by scanning the optical beam across the detector surface and sampling the detector response at 0.25-mm intervals. The detector signal was sampled and recorded at each position interval, and the data were normalized to the value of the response at the center of the detector. The results of the spatial uniformity measurements for the SWNT-coated detector are shown in Fig. 2. The results indicate that the variation in response over a 10-mm-wide detector area was approximately 1%.

In a similar manner, the directional uniformity was evaluated. But, rather than sampling the detector response in a two-dimensional planar array, the detector response at a single point located at the geometric center of the circular detector area was acquired. The probe beam from the laser was initially aligned to be centered and normal to the detector-aperture plane. For each field-of-view measurement

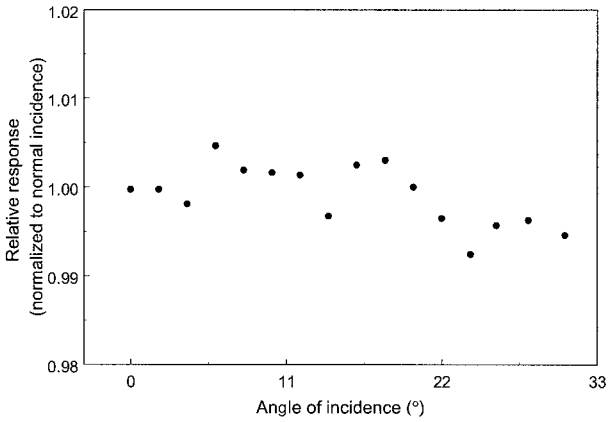

Fig. 3. Directional uniformity of the SWNT-coated detector acquired at 850-nm wavelength.

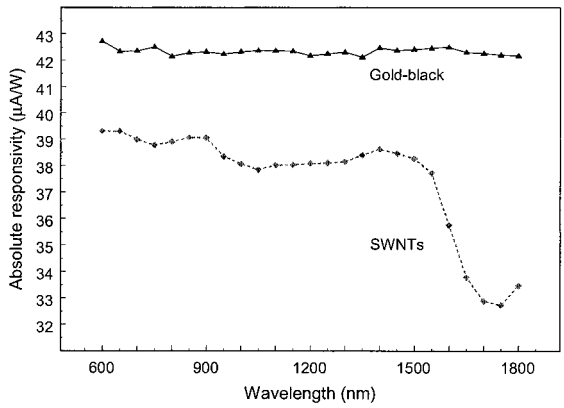

Fig. 4. Spectral responsivity of two pyroelectric detectors that were identical except for their coatings.

the laser was rotated away from normal incidence. The laser was incrementally revolved around the aperture center to define the probe-beam orientation. At each 2° increment, the detector response was sampled and the variation relative to the response at normal incidence was recorded. A surface map of detector response as a function of angle of incidence, in the presence of the SWNT coating, is shown in Fig. 3.

The measurement system for the spectral responsivity results was based on a lamp source, a grating monochromator, and a National Institute of Standards and Technology (NIST) transfer-standard detector.[11] The calibration procedure provided absolute spectral responsivity relative to a known NIST-calibrated transfer standard in 10-nm wavelength increments from 600 to 1800 nm. The NIST transfer standard was a pyroelectric wedge trap detector that was calibrated with the NIST C-series calorimeter.[12] The output beam from the monochromator (transmitted through air) was directed alternately onto the NIST standard detector and the test detector with a two-position mirror. The beam was focused to a beam size of approximately 2 mm × 2 mm, normal to the plane of the detector surface, with a bandwidth of 6 nm or less depending on the wavelength. Absolute spectral responsivity results are shown in Fig. 4.

4. Analysis and Comparison

The spatial and directional uniformity variations we observe are relatively small compared with our stated uncertainty. Therefore it is difficult to make rigorous conclusions about disadvantages of the SWNT coating. Small variations are generally desirable for detectors capable of measurements having low uncertainty such as we require for direct-substitution radiometry. By comparison, the large area photodiodes (3–5-mm diameter) that we might choose as an alternative for this wavelength range have spatial variations of 3% or more and directional variations that inherently depend on wavelength, angle of incidence, and polarization.[13] On this basis alone, a pyroelectric detector with nearly four times the area and spatial and directional response variations of approximately 1% is encouraging for this early work. Large area gold-black-coated pyroelectric detectors with comparable spatial and directional uniformity have been documented in the past; however the shortcomings of gold black, such as low damage threshold, remain.[4,6] Spatial and directional uniformity variations are wavelength dependent and a complete evaluation requires measurements at multiple wavelengths, which is beyond the scope of the present investigation.

For a greater understanding of the optical properties of carbon SWNTs, the absolute responsivity measurement is a useful diagnostic for several reasons. If the detector properties independent of the coating are well known, the absorptance of the SWNT coating can be measured. The optical reflectance can be calculated on the basis of specular absorptance at normal incidence rather than measuring a relatively small and diffuse reflectance of a coating sample alone. In the present case we have the advantage of an absolute responsivity measurement system with well-known and documented uncertainties.[11]

Based on our spectral responsivity measurement results and the modeling by Chen[1] and others,[2,3] we undertook a simple analysis of the absorptance of the SWNT-coated pyroelectric detector that begins with two dielectric functions, one describing a semiconductor and the other a metallic SWNT.[14] Both the dielectric functions and the critical values of the relaxation rates and charge carrier energy have been documented for semiconducting and metallic SWNTs.[1] Ugawa *et al.*[2] and separately Chen[1] gave the two dielectric functions as the Drude model

$$\varepsilon_m(\omega) = \varepsilon_\infty - \frac{{\omega_p}^2}{\omega^2 + i\gamma\omega} \tag{1}$$

for metal SWNTs and a Lorentzian model

$$\varepsilon_s(\omega) = \varepsilon_\infty - \frac{{\omega_p}^2}{\omega^2 - {\omega_o}^2 + i\Gamma\omega} \tag{2}$$

for semiconductor SWNTs, where ε_∞ is the electronic

Table 1. Summary of Properties for Eqs. (1)–(3)

Property	Value[a] (eV)
Electronic core contribution ε_∞	4
Relaxation rate of charge carriers $\hbar\gamma$	0.1
Relaxation rate of charge carriers $\hbar\Gamma$	0.14
Plasma frequency of charge carriers $\hbar\omega_p$	2.5
Center frequency $\hbar\omega_o$	5

[a]From Ref. 1.

core contribution, ω_p is the plasma frequency of charge carriers, ω_o is the center frequency, ω is the photon frequency, and γ and Γ are the relative relaxation rates of the charge carriers of the metal and semiconductor systems.

The optical properties of structurally inhomogeneous materials consisting of particles much smaller than the wavelength of light can be described by effective dielectric functions.[15] An effective medium approximation (EMA) has been employed in the past as a valid means of calculating a dielectric function of bulk SWNTs containing a mixture of metal and semiconductor SWNTs as well as for idealized vertically oriented carbon nanotubes.[3] We state the EMA equation here as

$$f\frac{\varepsilon_m(\omega)-\varepsilon(\omega)}{g\varepsilon_m(\omega)+(1-g)\varepsilon(\omega)}+(1-f)\frac{\varepsilon_s(\omega)-\varepsilon(\omega)}{g\varepsilon_s(\omega)+(1-g)\varepsilon(\omega)}=0. \quad (3)$$

The value of depolarization factor g has not been addressed in detail for SWNTs. Here we stop short of the curve fitting using the EMA. That is, we consider only results that we would expect if the SWNT–detector system behaved like a detector coated exclusively with either metal ($f = 1$) or semiconductor tubes ($f = 0$). The representative values in Table 1 used in Eqs. (1) and (2) are those given by Chen[1] following an evaluation of SWNT reflectance over a broad photon energy range.

We have gone beyond previous work by modeling the pyroelectric detector as the substrate of the SWNT coating and calculating the relative response of the detector, assuming the response is proportional to the absorptance of the detector and coating combination. The ideal performance was calculated from the index of refraction, $n(\omega) = \sqrt{\varepsilon(\omega)}$, obtained from Eqs. (1)–(3) and then modeling the optical absorptance of the detector and coating combination. The dimensions of the detector and coating thickness we modeled are identical to those of the detector we built, as described in Section 3. The $LiTaO_3$ thickness was 60 μm, the Ni electrode thickness was 25 nm (each face), and the SWNT coating thickness was 2 μm. The detector absorptance was calculated from 600 to 1800 nm at normal incidence. The detector coating was modeled by use of a commercially available optical coating design program as a convenient means of manipulating the Fresnel formulas.[16]

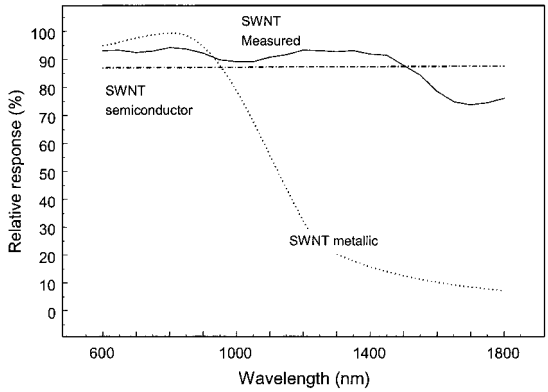

Fig. 5. Relative response of a SWNT-coated pyroelectric detector compared with predicted responses for films made exclusively of either semiconductor SWNTs or metal SWNTs. The detector model includes 25-nm Ni electrodes, 60-μm-thick $LiTaO_3$, and a 2-μm-thick SWNT coating.

We compared the measured performance of the SWNT-coated detector (in Fig. 4) with the ideal performance we would expect if the SWNT coating were either semiconductor or metallic. Based on our detector coating model and the wavelength range of interest, we assumed that transmittance through the coated detector is negligible. We also assumed that the relative detector responsivity is proportional to the optical absorptance.[4] The relative detector responsivity that we calculated is based on our knowledge of the absolute responsivity that we measured and the fact that the efficiency of the gold-black-coated pyroelectric detector has an absorption efficiency greater than 99%. The results of this comparison are shown in Fig. 5.

Two things are immediately apparent from this comparison. We expect a pyroelectric detector coated with semiconductor SWNTs to have a uniform spectral response, but to have only an 85% absorption efficiency. We would expect a similar detector coated with metal SWNTs to have a higher efficiency over a limited wavelength range but poor spectral uniformity at wavelengths greater than 900 nm. The performance of the detector that we actually measured is somewhere between these two cases. It is likely that this is because our SWNT coating is a mixture of semiconductor and metal tubes. Raman spectroscopy of these materials that employ both 488- and 633-nm excitation indicates that both semiconductor and metal SWNTs are present.[17] Also, the physical structure and roughness of the coating (in whatever tube form) could have some influence on the absorptance of the coating.

5. Discussion

Analysis of the optical properties of SWNTs by use of the EMA raises two questions and one new challenge: (1) Is it valid for a mixture representing portions of SWNTs that are semiconductor and metallic? (2) To

what extent is the topology of the SWNT coating important; that is, the roughness, physical structure, proximity, or entanglement of tubes with each other, compared with the crystal structure of the nanotubes? (3) Finally, (a grand challenge) if there is a specific chirality of SWNT that is preferable, can it be isolated? The results from Chen[1] and others suggest that the answer to our first question is yes. This is an important basis and point of departure for future study. We now have limited control over the type or proportion of species we produce by laser vaporization, although others have reported success in separating bulk forms of nearly homogeneous semiconductor or metal SWNT species.[18–20]

When we consider topology of the system and not just the material properties in the EMA, the depolarization term accounts for topology.[1,2] Ugawa *et al.*[2] merely state this term. Chen[1] acknowledges Ugawa *et al.*[2] but does not give details. In the past gold black[6] has been considered for which the matrix material is gold (which is highly reflective) but the structure is highly absorptive. Becker *et al.*[15] explored this in their model dielectric function of metal particles. They compared the spectral dependence of the absorption constant of gold-black layers with that of layers in which the particles are isolated from each other. Becker *et al.*[15] presented an EMA equation for gold black (a diffuse metal) with the following dielectric function:

$$\varepsilon(\omega) \approx 1 - \frac{fg_0\left(\frac{e}{\hbar}\omega_p\tau\right)^2}{1+\left(\frac{e}{\hbar}\omega\tau\right)^2} + i\,\frac{fg_0\left(\frac{e}{\hbar}\omega_p\right)^2\tau}{\frac{e}{\hbar}\omega\left[1+\left(\frac{e}{\hbar}\omega\tau\right)^2\right]}, \quad (4)$$

where e is the elementary charge constant, τ is the electronic collision time, and other variables are as defined in Section 4. To understand the role of topology it would be simpler first to consider a single tube species and adjust the g_0 percolation factor as necessary by use of a model similar to that of Eq. (4). Here we are not interested in just understanding the optical properties, rather our ultimate goal is to design detector coatings having high absorption efficiency. If we can understand and control the coating topology, we can objectively solve the coating design problem.

This leads us to address the most significant challenge. From our analysis, semiconductor tubes might be preferable because our calculations indicate the absorptance is high and flat in the visible and the near infrared. Future study will involve the examination of samples that have been enriched in semiconductor SWNTs. Additionally, for metal or semiconductor tubes, the absorptance might be improved by first understanding how to control the coating process, defining the coating topology as necessary and perhaps employing thicker coatings. It is also perhaps possible that a semiconductor tube of a specific chirality will provide the optimal coating. Isolation or synthesis of a single chirality phase of a pure bulk sample has not yet been achieved.

Table 2. Summary of Thermal Properties of Various Coatings Suitable for Thermal Detectors

Property	Material	Value	Reference
Thermal conductivity (W/mK)	SWNT	100 to 1000	21
	Gold black	0.03	30
	Carbon black	0.003	30
Specific heat (mJ/gK)	SWNT	600	31
	Gold black	130	22
Damage threshold (mJ/cm^2)	MWNT	547	32
	Gold black	38	6
	Commercial thermal detector (ceramic)	250	33
	Ni on Cu, Ni on sapphire	150	6

6. Summary of Thermal Properties

In Table 2 we briefly provide a summary of thermal properties from the literature that are relevant to coating design. The thermal conductivity as reported by Berber *et al.*[21] is exceptional and distinguishes carbon nanotubes from available alternatives. The specific heat of SWNTs is also relatively high. The combination of high thermal conductivity and high specific heat is an important consideration for coatings used in electrical substitution or other schemes for which the electrical inequivalence would compromise the accuracy of the device to which it is applied, such as the electrically calibrated pyroelectric,[22] cryogenic radiometer,[23] or isoperibol calorimeter.[24]

So far we have informally assumed that laser energy absorbed by a semiconductor SWNT is converted to heat by phonon relaxation. It has been shown that SWNTs can behave as a saturable absorber,[25] which suggests that the absorption coefficient changes depending on the laser energy and wavelength. It has also been shown that carbon SWNTs generate photons in studies of photoluminescence.[26] Either way, this behavior complicates the applicability.

Future investigation of the thermal properties of carbon nanotube coatings might employ techniques described by Bauer and Ploss[27] or Phelan and Cook,[28] whereby the specific heat and thermal diffusivity are determined from the volume average of the change in temperature with respect to time for a pyroelectric film. In addition to considering the thermal properties of the detector and substrate material, the coating properties can be determined if properties of the detector and coating are well known.

7. Conclusion

We have built and evaluated a pyroelectric detector coated with SWNTs. The spatial and directional uniformity measurement results, which indicated variations of approximately 1%, are favorable compared with other alternatives. We have studied the optical properties of SWNT coatings based on an EMA model and our spectral responsivity measurement results

that document responsivity variations as large as 15%. Metallic SWNTs have a spectral behavior that is more uniform but less efficient than the coating we measured. The EMA also shows that semiconductor tubes have greater efficiency over a limited wavelength range but are less spectrally uniform than metallic tubes. In the future, the EMA described by García-Vidal *et al.*[3] and by Becker *et al.*,[15] whereby the topology is explicitly considered, might be exploited for the design of either SWNT or multiwall nanotube-(MWNT)-based coatings. Thermal properties summarized from the literature indicate that carbon-nanotube-based coatings warrant further investigation and could be superior to some current alternatives such as carbon-based paints and gold black. In the future, we plan to examine SWNT coatings that are enriched in semiconductor SWNTs.[18] Also a hot wire chemical vapor deposition process that produces graphitic MWNTs virtually free from nonnanotube carbons[29] might be adapted to create thermal detector coatings that could further our understanding of the optical, electrical, and thermal properties of MWNTs.

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33. This is a typical value as given by one or more vendors (not specified) and depends on the nature of the laser pulses and the mode of cooling the detector.