Characterisation of a new carbon nanotube detector coating for solar absolute radiometers

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Abstract. A new sprayable carbon nanotube coating for bolometric detectors aims to increase the absorptance compared to regular space qualified black paints. In collaboration with the National Institute of Standards and Technology (NIST), we have characterized the optical properties and mechanical and thermal stability of the carbon nanotube coating inside conical shaped cavity detectors.

1. Introduction

We characterized three cavity detectors coated with spray-on carbon nanotubes by Surrey NanoSystems® [1]. The cavity detectors are supposed to be used in the Digital Absolute Radiometer (DARA) developed and built for the PROBA-3 satellite. The cavities are made of silver with a gold coating layer on the exterior face, to suppress radiative heat exchange with the surroundings (e.g. [2]).

The detectors were evaluated under two different experiments. The first cavity was exposed to a high-power UV laser to study the accelerated ageing of the carbon nanotubes under optical conditions similar to 1 year of Total Solar Irradiance (TSI) space measurements. The second cavity was tested in a thermal cycling test chamber simulating thermal variations onboard a satellite. The third one served as a reference and as such was not subjected to any test. We studied the reflectance of the detectors and evaluated the coating qualitatively by Scanning Electron Microscopy (SEM) before and after the tests to evaluate the performance of the coating and to identify differences in the absorptance and in the structure of the carbon nanotubes.

2. Reflectance measurements

Reflectance maps of the cavities were produced in the optics laboratory at PMOD/WRC (Figure 1) using a setup with an integrating sphere as described in [2, 3]. Two laser wavelengths (633 nm and 375 nm) were used to study the spectral response of the coating. Both lasers have a stable output power of approximately 5 mW.

The same cavities were subjected to reflectance measurements in the Laboratory for Atmospheric and Space Physics (LASP) (Figure 2) facilities at 406 nm, 520 nm, 660 nm, 830 nm, 1310 nm, and 1625 nm.
Figure 1. - Example of a measurement done at PMOD/WRC (Cavity 1 – 633 nm).

Figure 2. - Example of a measurement done by NIST (Cavity 1 – 660 nm).

Figure 3. Summary of reflectance measurements before the tests.

The reflectance measurements performed at PMOD/WRC agree well with the measurements performed with a similar integrating sphere setup at LASP as can be seen in Figure 3. These reflectance values were used as a reference to compare them with the reflectance measurements after the tests and will give us information about potential degradation occurring in the cavities because of the thermal cycling or the UV treatment.

The discrepancy in reflectance shown for Cavity 2 in contrast to 1 and 3 is attributable to Cavity 2 having only one layer of carbon nanotubes against the two layers in the remaining cavities. Three layer coatings were not tested, but a third layer would not improve the reflectance values according to the manufacturer.
3. Scanning Electron Microscope

In addition to the reflectance measurements, SEM imaging provides a qualitative study of the carbon nanotubes to identify any coating defects inside the cavities. Before and after the cavity treatment (either the ageing or the thermal cycling), SEM pictures of the surface of the coating were taken for a qualitative impression of how the mentioned treatments affect the surface of the sprayed paint and the structure of the carbon nanotubes.

![Figure 4. SEM pictures of Cavity 1 (left: pre-treatment; right: post-UV treatment). No significant differences are visually detected.](image)

![Figure 5. SEM pictures of Cavity 2 that remains as a reference (left: pre-treatment; right: post-treatment of the other cavities). Defects in the walls of the cavities attributable to a wrong handling of the detector, which results in a higher reflectivity.](image)

Because of the binder used in the cavities, a commercial glue that fixed the heater foil inside the cavity before applying the carbon nanotubes coating, the process temperature for spraying the carbon nanotubes was limited, obtaining the meniscus as a result that can be seen in Figures 4 and 6. This does not represent a problem in the properties of the detector, but it is something to be optimized by looking for components with a higher temperature resistance. The currently used binder for the heater foil only allows temperatures up to 120°C, whereas the typical application temperature for the sprayable CNT’s is 280°C.
4. Thermal cycling
Thermal cycling was performed for Cavity 3 to evaluate any gross deficiencies in the assembly (including both coating and substrate) preliminary to eventual space qualification testing. Our radiometer was subjected to thermal cycling for 21 cycles over 6 days. Each cycle was 6 hours and 30 minutes, with a maximum temperature of 80 °C and a minimum temperature of -20 °C.

The cycle was thermally controlled by a thermistor inside the chamber every minute to check the stability of the test.

This preliminary thermal cycling was not seen to induce coating cracks and with their respective reflectivity changes. The cavity structure demonstrated no degradation.

5. UV exposure
Cavity 1 was subjected to a high-power UV laser ($\lambda = 248$ nm) as our goal was to simulate the equivalent UV dose for one year of full time solar exposure. The setup, shown in Figure 7, has a 0.8 cm diameter aperture to limit the UV laser beam diameter and the sample was installed inside a vacuum chamber maintained below $6 \times 10^{-4}$ mbar and the cavity was thermally controlled by a thermistor with a maximum temperature of 85 °C.

A beam splitter reflected 3.8% of the incident radiation to a monitor detector and allowed us to have a real-time measurement of the pulse.

The cavity radiometer was exposed during 5 hours to 3.6 million pulses of approximately 20 mJ/pulse with a frequency of 200 pulses/s, with an average irradiance of 2 W/cm².

First, we crudely estimate the temperature rise that might show in the carbon nanotubes due to the pulsed laser by assuming 1) each pulse is absorbed by the nanotubes only without conduction to the substrate, 2) the absorbed energy propagates to the substrate before the arrival of the next pulse, and 3) the substrate temperature rise is trivial. The temperature rise of the nanotubes is estimated, using the familiar heat capacity formula:
\[ E = m \cdot C \cdot \Delta T \]  

(1)

Here \( m \) is the mass of the carbon nanotubes in the cavities with two layers; 5 mg according to the manufacturer, the specific heat capacity \( C \) can be compared for the value in [4] for MultiWall carbon nanotubes (MWCNT) and is \( 8 \text{ J/mol K} \) and the energy of the laser \( E \) is 20 mJ. Using these values in (1) we find a temperature rise of 6 K for each laser pulse for the carbon nanotubes. This value suggests that the peak of temperatures during each pulse will increase the temperature, and, as it is indicated by the manufacturer, a temperature higher than 325°C would have broken the hydrophobic protection film, consequently damaging the coating and increasing the reflectivity.

6. Results and Discussion

After the different treatments, we again measured the reflectance of the cavities and we compared them to the measurements before the treatments in Figure 8.

![Figure 8. Comparison of the three cavities previously and after each treatment by LASP.](image)
Cavity 2, which served as a reference, showed a decrease in the reflectance values even though
no treatment was applied. This discrepancy is most likely attributable to experimental errors during the
measurements after the treatments, smaller than 10%, described in [3].
Measurements of Cavity 3 before and after thermal cycling show no significant difference and indicate
the thermal cycling has not affected the carbon nanotubes within the uncertainties shown for Cavity 2.

Although there is no significant difference in the SEM imaging (see figures 4 to 6), after UV laser
accelerated aging, Cavity 1 suffered a significant increase in the coating reflectance, as can be seen in
Figure 8. The temperature reached in the carbon nanotubes is most likely higher than what we measured
in the cavity and might be above the temperature limit of the hydrophobic film. This overheating of the
coating might induce breaks in the protection film and damage on the carbon nanotubes. We saw this
behaviour, extremely pronounced, for a VACNT sample that had a thermal grease under the substrate.
This effect will be discussed in another paper on, currently under development.

Another explanation could be outgassing produced by the carbon nanotubes coating inside the vacuum
chamber which might have contaminated the carbon nanotubes.

For space applications, measuring TSI, the nanotubes will not be subjected to high UV pulse peaks, as
we performed in the experiment. Therefore, the temperature in the carbon nanotubes will not be higher
than the binder limit.

As well, outgassing of the detector will be performed before launching, thus potential contamination
will be included.

On the other hand, we proved that thermal changes may not affect the detectors.

We performed the first steps in the characterization of this novel carbon-nanotube coating in a conical
cavity for TSI. Further optimization of the detectors, such as a binder with higher temperature resistance
or a new cavity shape optimized for the characteristics of the carbon nanotubes, may be applied. As
well, further experiments for a better knowledge of the outgassing or analysis of the behaviour under
environment with atomic oxygen will give us more information about the application of this new
detectors for TSI.

7. References

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Acknowledgments

This work is supported by SNF grant 200021 162926.
Thanks to the Sources and Detectors Group of NIST in Boulder (Colorado), specially to Michelle
Stephens, Brian Simonds, Chris Yung, David Livigni, and John Lehman for opening their laboratories
to me, and for investing their time and effort for this work.