# Temperature and Pressure Dependence of the Reflectivity of Vertically Aligned NanoTube Arrays

Martin Dury<sup>1</sup>, Nicole George<sup>1</sup> and Nigel Fox<sup>1</sup>

<sup>1</sup> National Physical Laboratory (NPL), Teddington, United Kingdom Corresponding e-mail address: martin.dury@npl.co.uk

The very high absorptivity of Vertically Aligned NanoTube Arrays (VANTA) coatings makes them desirable for primary radiometry. While their reflectivity is typically measured at ambient temperatures and atmospheric pressures, when applied in cryogenic radiometry they are cooled to very low temperatures and very low pressures. It is therefore essential that the reflectance of VANTA does not increase with decreasing temperature such that the change in cavity reflectance becomes a dominant measurement uncertainty. This paper reports preliminary measurements showing a <13 % increase in reflectivity at 633 nm between room and cryogenic operating temperatures for a VANTA coating applied to a copper substrate. Such a change in coating reflectance approximates to a change in cavity reflectance of 3 ppm for a typical NPL cryogenic radiometer cavity.

# **INTRODUCTION**

The reflectance of VANTA is known to be very low across the spectral range from visible to infrared wavelengths [1-8], and is usually measured at ambient temperatures and at atmospheric pressures. The very high absorptance properties of VANTA therefore make it an attractive material for use in primary radiometry, particularly in cryogenic radiometry [9]. As the detectors in cryogenic radiometers are typically cooled to temperatures within the range from 14 K to 80 K and are operated at pressures of the  $10^{-7}$  mbar level, it is important that the reflectance of VANTA coatings does not increase significantly, leading to it becoming a dominant uncertainty. measurement The target cavity reflectivity for NPL cryogenic radiometers is  $\rho < 30$ ppm [10].

NPL is currently developing primary radiometry for the Traceable Radiometry Underpinning Terrestrialand Helio-Studies (TRUTHS) mission [10]. This mission aims to provide a means of calibrating Earth Observation (EO) satellites through the measurement of reference targets. For the mission to be a success it is essential that the behaviour of VANTA coatings is well characterised for use in space environments. This paper describes an investigation to simulate the space environment that will be endured by the VANTA coatings used in the detector cavities for the TRUTHS mission, through characterisation of their temperature and pressure dependence on reflectivity.

#### **EXPERIMENTAL SETUP**

A variable cryostat was used to measure the temperature and pressure dependence of the reflectance of VANTA. Figure 1 presents a schematic of the experimental setup. The cryostat was modified such that a VANTA coated copper sample plate could be mounted within. A 25 mm diameter integrating sphere was attached in front of the VANTA sample. This admitted light from a HeNe 633 nm laser through the window at the front of the cryostat and through the sphere's front port onto the sample via an additional port at the back. A silicon photodiode mounted to a side port on the sphere monitors the amount of reflected hemispherical radiation. A beamsplitter and additional photodiode outside of the cryostat measured the optical power stability of the laser beam, allowing for the correction in optical input power variations.



Figure 1 Schematic of the experimental setup.

Variations in the performance of the integrating sphere and the silicon photodiode due to the changes in temperature and pressure were accounted for. To evaluate potential changes in the setup, the integrating sphere was oriented such that the first reflection of the laser beam was from its internal barium sulphate coating. First the change with pressure was measured: the turbo pump was switched on and the signal from the sphere mounted photodiode was monitored as the pressure dropped to it lowest level at room temperature. The cryostat was then cooled to cryogenic temperatures and the signal from sphere mounted photodiode recorded as cryopumping was achieved.

## RESULTS

The temperature dependence of the optical setup was measured to be less than  $\pm 1.5$  % over the temperature range of interest. This small percentage has such a negligible effect on the results of this study it is accounted for as a measurement uncertainty.



**Figure 2** Preliminary data showing temperature dependence of reflectivity of the VANTA sample. Error bars (95% confidence) account for temperature dependence of setup.

Figure 2 shows preliminary data for the temperature dependence of the VANTA sample, which has been corrected for drift in laser power. The data suggest that the reflectivity of VANTA (at 633 nm) increases by <13% between room and cryogenic operating temperatures.

## CONCLUSIONS

The low spectral reflectance of VANTA coatings makes them attractive for use in cryogenic radiometer detecting cavities. Reflectance measurements are typically performed at ambient temperatures and at atmospheric pressures. As cryogenic radiometers operate at near absolute zero temperatures and almost vacuum pressures it is important that the reflectivity of VANTA does not increase significantly when operating under these conditions.

Preliminary measurements of VANTA applied to a copper substrate suggest that the reflectivity of VANTA (at 633 nm) increases by <13% between room and cryogenic operating temperatures, which represents a change in cavity reflectance of approximately 3 ppm for a typical NPL cryogenic radiometer cavity. The effect would need to be 30 times greater to become a dominating uncertainty in cryogenic radiometer measurements.

This study is currently limited to a single wavelength. Further work investigating the change in reflectivity at different wavelengths from UV to NIR range and with decreasing pressure are to be reported at NEWRAD 2020.

## ACKNOWLEDGEMENTS

The authors wish to acknowledge the work of Rainer Winkler who performed initial NPL investigations into the reflectance properties of VANTA.

#### REFERENCES

- 1. Yang ZP, Ci L, Bur JA, Lin SY, Ajayan PM. Experimental observation of an extremely dark material made by a low-density nanotube array. Nano Lett. 2008;8(2):446–51.
- Mizuno K, Ishii J, Kishida H, Hayamizu Y, Yasuda S, Futaba DN, et al. A black body absorber from vertically aligned single-walled carbon nanotubes. Proc Natl Acad Sci U S A. 2009;
- Quijada MA, Hagopian JG, Getty S, Kinzer, Jr. RE, Wollack EJ. Hemispherical reflectance and emittance properties of carbon nanotubes coatings at infrared wavelengths. In: Cryogenic Optical Systems and Instruments XIII. 2011.
- 4. Yang ZP, Hsieh ML, Bur JA, Ci L, Hanssen LM, Wilthan B, et al. Experimental observation of extremely weak optical scattering from an interlocking carbon nanotube array. Appl Opt. 2011;
- 5. Theocharous E, Deshpande R, Dillon AC, Lehman J. Evaluation of a pyroelectric detector with a carbon multiwalled nanotube black coating in the infrared. Appl Opt. 2006;
- 6. Theocharous SP, Theocharous E, Lehman JH. The evaluation of the performance of two pyroelectric detectors with vertically aligned multi-walled carbon nanotube coatings. Infrared Phys Technol. 2012;
- Chunnilall CJ, Lehman JH, Theocharous E, Sanders A. Infrared hemispherical reflectance of carbon nanotube mats and arrays in the 5-50 μm wavelength region. Carbon. 2012.
- 8. Lehman JH, Lee B, Grossman EN. Far infrared thermal detectors for laser radiometry using a carbon nanotube array. Appl Opt. 2011;
- Tomlin NA, Lehman JH. Carbon nanotube electricalsubstitution cryogenic radiometer: initial results. Opt Lett. 2013;
- 10.Winkler R. Cryogenic Solar Absolute Radiometer A potential SI standard for Solar Irradiance. University College London; 2012.
- 11. TRUTHS NPL [Internet]. [cited 2020 Jan 21]. from: https://www.npl.co.uk/earth-observation/truths