

Effects of Atomic Oxygen and UV rays ageing on the Reflection Coefficient of Carbon/Carbon Plates in the 12-18 GHz Frequency Range

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Abstract— Advanced carbon-based ceramic materials, such as Carbon/Carbon (C/C) are commonly employed in aerospace industry to face the thermo-mechanical stress suffered by the spacecraft external structures during re-entry in earth atmosphere. Thanks to very low thermal expansion and outstanding chemical stability at extreme temperatures, in fact, such materials represent the ideal candidates for re-usable space integrated systems, such as TPS, nozzles and ablation thermal-proof structures. In long-time missions, on the other hand, issues related to continuous and extended exposure of the spacecraft surfaces to the detrimental space environment must be carefully considered. In this frame, also the tracking of satellites by mean of radar systems is a vital aspect for a safe stay in space. In LEO working conditions, the combined effects induced by severe thermal cycles, ultra-high vacuum and UV/Atomic Oxygen(AO) irradiation can be of impediment for the use of C/C as re-entry protection material, since a significant surface oxidation leads to so high TPS damaging as to compromise its main functionality. This surface oxidation can also affect the radar visibility of satellites. Aim of this paper is to evaluate the effects of the AO/UV ageing on C/C plates reflection coefficient, in order to avoid malfunctioning in the radar visibility.

Keywords—Atomic Oxygen, Reflection Coefficient, Space Environment

I. INTRODUCTION

The optimization of the satellites lifetime is at present day one of the most important tasks to face when a satellite is designed. In order to face severe thermal cycles, ultra-high vacuum and UV/AO irradiation, it is necessary to protect the spacecraft to guarantee an enhanced lifetime. Beside the protection of the spacecraft itself during space missions, on the other hand, that is very important for limiting the space junk and guarantee a safe space. In this frame the tracking of satellites and spacecraft and the determination of their attitude by mean of radar systems and lightcurve method [1-6] appears as one of the most relevant activities in order to monitor and control the spacecraft motion during its orbit.

Nowadays, despite a copious scientific production on space environment effects and on radar systems, a few works

have been performed in order to correlate the space environment effects with the radar visibility of space systems. The use of C/C is suggested by its outstanding properties in terms of very low thermal expansion, low density, great chemical stability at high temperatures and good thermal conductivity. Despite these very good properties, the long exposure to the extreme conditions of the space environment shall give rise to several problems, especially oxidation and corrosion of the exposed surface. This degradation can lead to a decrease of the electromagnetic reflection properties, causing a lower radar visibility.

II. MATERIALS AND METHODS

A. Materials

A commercial C/C slab (from Mitsubishi Chemical Carbon Fiber & Composites, thickness ~6 mm, nominal density 1.437 g/cm³), selected for the production of a Space Environment Plate Protection System (SEPPS) to be mounted on a cube-sat, was cut by water-jet technique into square plates of side 100 mm (Fig. 1); from the same batch several samples have been obtained for the space environment characterization – these are labeled as C/C-plate.

C/C composites offer a wide range of properties that can be tailored by the selection of constituent materials, fiber orientations, and details of fabrication. Typically, C/C material and components are designed simultaneously so that the composite properties can be addressed to enhance the component performances. The characteristics of interest are strength and stiffness, fracture toughness, frictional properties, thermal conductivity and resistance to oxidation at high temperatures [7-9]. The operating mechanisms for these properties are quite different, especially in such multiphase composite materials. The physical characteristics of the constituents and their volume fraction, bonding and crack propagation mechanism, control the mechanical properties of the composite, whereas the thermal behavior is governed by thermal transport phenomena.

Moreover, the constituents - both reinforcement and matrix - are likely to undergo physical/chemical changes during processing, as influenced by heat treatment

temperature, differential dimensional changes, thermal stresses, and so on; all these factors determine the ultimate properties of the composite [10].

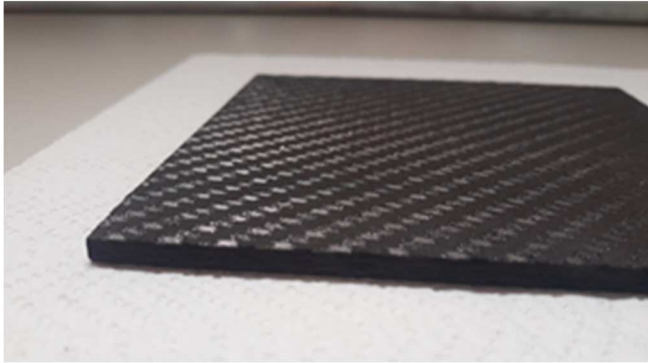


Fig. 1. Carbon/Carbon Plate

B. Atomic Oxygen/UV ageing

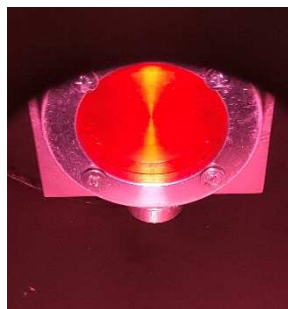
The atomic oxygen ageing simulator available at DIAEE LSA consists in a small vacuum chamber where a RF plasma source (Oxford Scientific Instruments - SPECS) is installed to provide a neutral oxygen plasma for the simulation of AO/UV effects on space materials in LEO.

The chamber has length of 52cm and has inner diameter of 16cm. A scroll XDS5 Varian Turbo V-550 is used to reach ultra-high vacuum conditions (minimum pressure achievable: 10^{-5} Pa). The working principle is based on the dissociation of the molecular oxygen flowing within the chamber, controlled by high precision flowmeters, by means of energy from a radio frequency source at 13.56MHz.

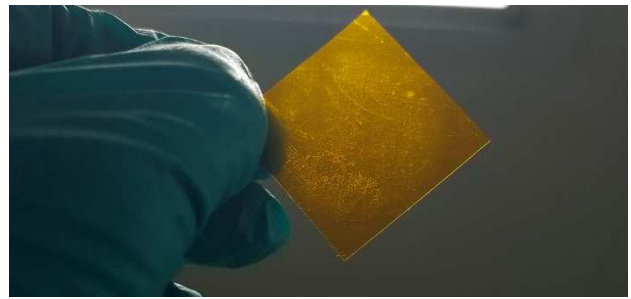
Such procedure gives rise to an oxygen-based beam constituted by 99% of neutral species (by about 60% of monoatomic O and 40% of molecular O_2) at a fluence of 8.7×10^{20} n.s./ cm^2 , and a remaining 1% of O^+ ions, with energy 5 ± 25 eV. The value of fluence is based on the erosion of a witness sample of Kapton HN (Fig.2), a material of known in-space erosion yield, as suggested by ASTM E2089. The working pressure is in the order of 10^{-4} Pa. For the weight measurements the experimental apparatus is equipped by a high precision micro-balance (Mettler-Toledo, sensitivity 0.002 mg).



(a)



(b)



(c)

Fig. 2. Kapton sample: in picture (a) the sample before the test; in picture (b) the sample during the test; in picture (c) the sample after the test. Note the central exposed zone.

In addition, an UV rays generator is used in order to study the synergistic effects of UV and AO. The UV generator is a highly stable mercury-xenon lamp (Hamamatsu - model LC8 Lightning Cure). The system provides high-intensity UV line spectra with an elliptical reflector (UV cold mirror) having reflectivity higher than 90% in the UV range and a quartz light guide with UV transmittance. The lamp works in horizontal position in order to have an optical system with low light loss. The spectral emittance field range is 200 to 600 nm with a maximum emission value at 365 nm. The radiation intensity of the lamp system is 410 mW/cm^2 (around 10 Suns) at 60 mm distance with an aperture size of 20 mm.

The whole facility is depicted in (Fig.3); the test set-up is the following [11-13]:

- First of all, the witness Kapton HN sample was previously tested in order to evaluate the Fluence. The Erosion Yield, indeed, is known for Kapton HN, so a reverse process is needed for the calculation of the AO effective Fluence. The calibration process takes a very long time, as the sample is tested and weighted in different steps, in order to have an "exposure table" with the different fluences related to the different times of exposure. In this paper the energy was set to 20 eV and the effective fluence reached was 8.7×10^{20} n.s./ cm^2 .

- The samples of materials under investigation were weighted before the test for the total mass loss evaluation, then they have been positioned inside the AO chamber and conditioned for 48h at a pressure of 260mbar as required by ASTM E2089 for AO tests.

- After such treatment, the samples have been exposed to AO, till a final exposition of 3000 Equivalent Sun Hours (EHS) at an effective fluence of 8.7×10^{20} n.s./ cm^2 , in the same conditions of the calibration. Then, the UV lamps were turned on to reach a samples exposure time of 3000 Equivalent Sun Hours at 410 mW/cm^2 . The working pressure was 3×10^{-4} Pa.

In Table 1 the test conditions and the erosion rate calculation sequence are indicated. In order to evaluate the erosion rate (E_y) of the samples, the first step is the evaluation of the mass loss, i.e. the loss of mass (ΔM) due to the AO exposure. After this step, the erosion yield can be easily calculated and given in terms of volume eroded for impacting oxygen atom, by knowing the values of fluence (F), density (ρ), and exposed area (A).

TABLE I. AO TEST CONDITIONS AND CALCULATION SEQUENCE OF THE EROSION RATE.

Calculation of Erosion Yield [cm^3/atom]	
AO Fluence	Total Mass Loss (TML)
$\sim 8.7 \times 10^{20} \text{ atoms/cm}^2$	$\Delta M / M$
$F = \Delta M / (\rho \cdot A \cdot E_y) \rightarrow \text{AO react coeff. (E}_y)$	

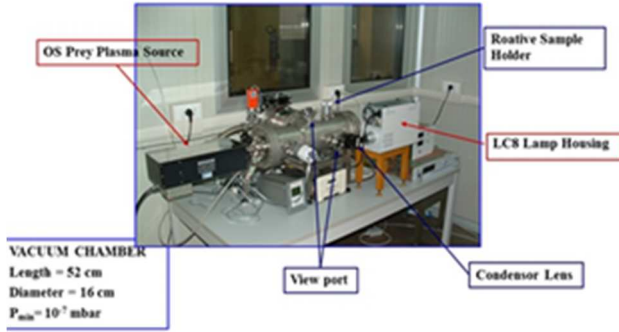


Fig. 3. Atomic Oxygen and UV facility

C. NRL Arch system for Reflection Coefficient

NRL Arch bi-static method is the industry standard for testing the reflectivity of materials. Originally designed at the Naval Research Laboratory (NRL), the NRL Arch enables fast, repeatable, and non-destructive testing over a wide range of frequencies [12-16]. Reflectivity, as described for all the different EM reflection evaluation methods [17], is defined as the reduction in reflected power caused by the introduction of a material, comparing to the “perfect” reflection which comes very close to what happens with a flat metallic plate. Measurements of a known sample consisting in a metal plate have been performed in order to be aware about errors in the NRL measurement set-up. The level of confidence of the measure was within 2dB interval, with respect to the reflection properties of the metal plate.

The antennas can be positioned anywhere on the arch to allow performance measurements with angles of attack not normal to the sample under test. In this paper, a 45° angle for each antenna has been chosen. The vector network analyzer is used for measurements to provide both stimulus and measurement, with one of the antennas that functions as a signal generator, and the other one as signal detector. After the calibration and the set of the reference point measuring the reflection of the metallic plate, the material under test is placed on the metal plate and the reflected signal is measured in dB. Time domain gate and anechoic panels are used to eliminate antenna cross-talk and clear the error otherwise introduced by room reflections. Using this configuration, it is possible to characterize the properties of systems in different directions.

The measurement system is based on Agilent 8571E software (material measurement) and the Agilent PNA-L N5230C vector analyzer. The antennas are Q-par Angus Ltd working in the range 12-18 GHz. The sample rating was set at 512 points, with a power of -15dBm and a bandwidth 1 kHz. The system is shown in Fig. 4. For each sample two measures are performed for the two different polarization of the antennas (TE and TM). The measurements reliability lies within the 2 dB range with respect to the reflection properties declared in the ECCOSORB data sheet.

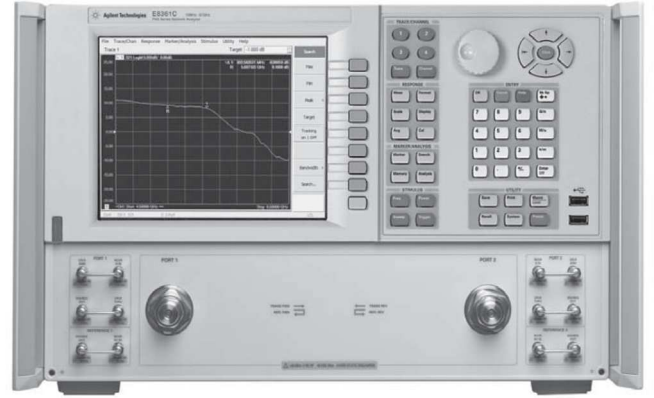


Fig. 4. The NRL Arch system: above, the arch and the antennas; below the Vector Network Analyzer.

III. RESULTS AND DISCUSSION

The test chain is defined as follows:

First of all, reflection measures have been performed on the C/C plate, achieving the reflection values for a “virgin” plate. Then the AO/UV test is performed on the same plate, achieving the Mass Loss and the Erosion Yield. Finally a second NRL arch test is performed on the same plate in order to establish if and how the AO and UV affected the sample in terms of Reflectivity values.

A. NRL Arch measures on virgin samples

In the following plot (Fig.5), the TE and TM polarization plot is shown. As expected, the reflection coefficient, given in dB by mean of the Scattering parameter S_{12} is a little bit lower than that of the reference metallic plate. That means a quasi-complete reflection of the material. In Fig. 6 the virgin sample surrounded by anechoic panels is show.

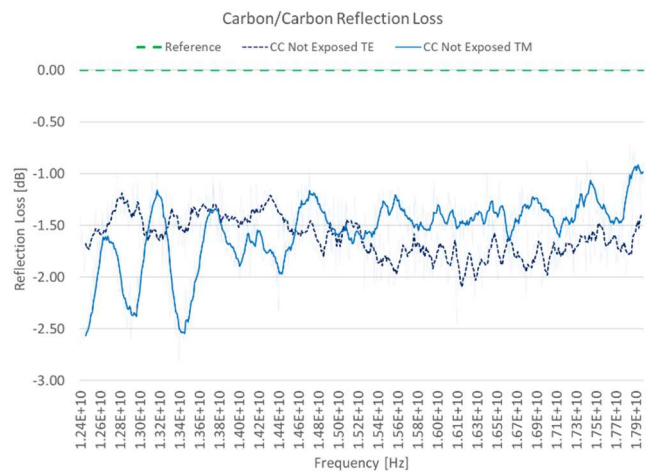


Fig. 5. The TE/TM polarization value of the Reflection Coefficient for a virgin C/C plate.

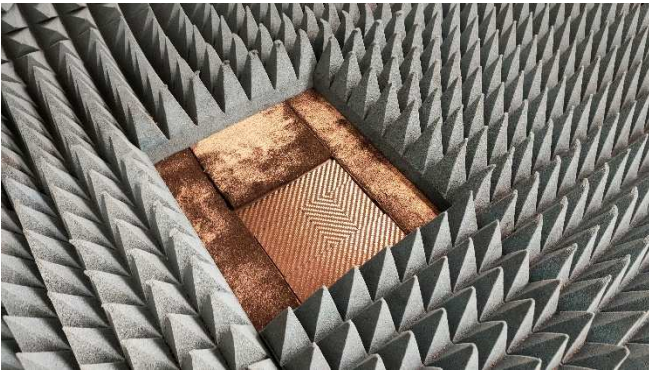


Fig. 6. The virgin sample under testing surrounded by anechoic panels

B. Atomic Oxygen and UV on C/C plate

The AO/UV test campaign provided the following parameters in terms of ΔM and E_y , as synthetized in Table 2.

TABLE II. AO TEST RESULTS.

Erosion Yield and ΔM	
ΔM [g]	E_y [cm^3/atom]
0.00025	9.1E-25

In Fig.7 some pictures of a C/C sample during the AO and UV test is given. It is worth to be noticed that both the Erosion Yield and ΔM are in line with the results obtained in previous works [11].

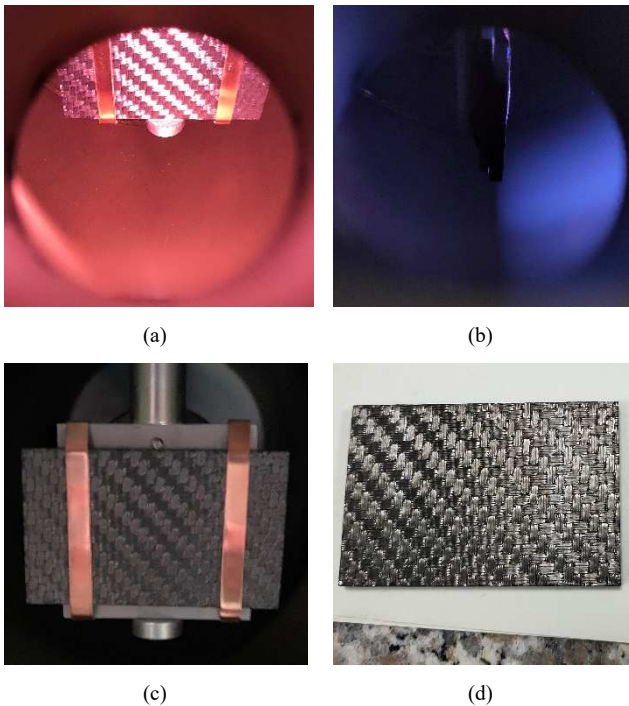


Fig. 7. C/C sample during AO+UV ageing test. In picture (a) the AO test; in picture (b) the sample during the UV ageing; in picture (c) the sample before all tests and in picture (d) the sample after the tests: note the slightly dark/brown spot on the center

C. NRL Arch measures on tested samples

In the following Fig.8 the TE/TM polarization plot is given for the AO-conditioned C/C material in respect to the virgin one. In this case the reflection coefficient, given in dB by mean of the Scattering parameter S_{12} , is affected by the oxidation products created by the AO/UV ageing test, with an absorption

peak at 12.9 and 1.35 GHz for TM polarization and a general absorption trend that is much higher than in the previous case.

In fact, despite the shape of the two polarization can be considered the same for both the conditions, there is a mean difference between them of 1.75 dB. It can be also noted that the absorption behavior at the absorption point of 13.5 GHz for the exposed C/C plate, involves a larger frequency span than the one of the not exposed plate, and it can be due to the absorption properties of the oxidation products. In Fig. 9 the sample under the arch is shown

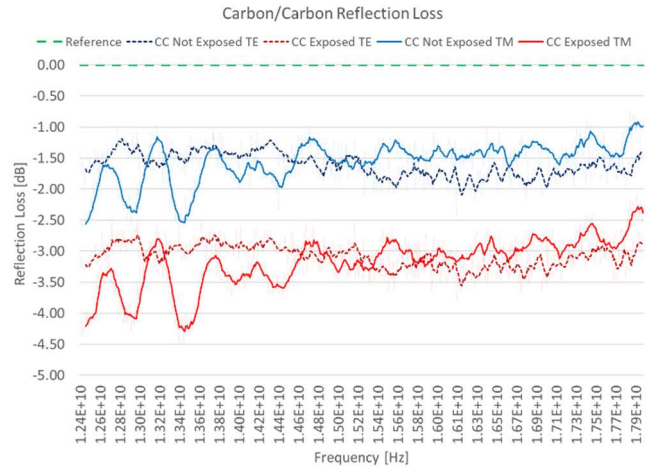


Fig. 8. The TE and TM polarization value of the Reflection Coefficient for the AO/UV tested C/C plate in respect to the virgin plates.



Fig. 9. The exposed sample surrounded by the anechoic panels

IV. CONCLUSIONS

In this paper the effects of AO/UV ageing on the reflection coefficient of C/C plates have been evaluated. The test campaign led to the conclusion that the oxidation products given by the ageing test (especially AO) have worsened the near perfect reflection properties of a C/C plate. The sample indeed was exposed to a AO flux with a fluence of 8.7×10^{20} n.s./ cm^2 , for an equivalent time of 3000 EHS and to a UV Flux with an intensity of $410 \text{ mW}/\text{cm}^2$ at 365 nm. A darkening of the sample was noticed on the exposed zone, with dark grey/brown tones that witness the ageing action given by the AO/UV fluxes and a mass loss of 0.00025 g, with an Erosion Yield of $9.1 \times 10^{-25} \text{ cm}^3/\text{atom}$. The result in terms of reflection properties was a general lowering of the reflection coefficient and two main absorption peak at 12.8 and 13.5 GHz for the TM polarization. The peak at 13.5 GHz is the main difference between the virgin and the exposed sample, as, for the exposed sample, it involves a larger frequency span than the one of the not exposed plate. As said that can be related to the AO scratching action on the C/C samples that creates

inhomogeneities on the surface and to the chemical reactions that occurs on the exposed surface, with the creation of oxidation products. For the evaluation of the reflection properties the NRL arch method was used, normalizing the samples measurements in respect to a reference metal plate, which represents the perfect reflection. The next step of the research will be to evaluate the same effects on C/C plates coated with protective layers against oxidation.

REFERENCES

- [1] F. Piergentili, R. Ravaglia, F. Santoni, "Close approach analysis in the geosynchronous region using optical measurements", *Journal of Guidance, Control, and Dynamics*, Volume 37 (2), pp. 705-710, 2014.
- [2] F. Santoni, F. Piergentili, "UNISAT-3 attitude determination using solar panel and magnetometer data", *Proceeding of the International Astronautical Federation - 56th International Astronautical Congress*, Volume 5, pp. 2812-2819, 2005.
- [3] T. Cardona, P. Seitzer, A. Rossi, F. Piergentili, F. Santoni, "BVRI photometric observations and light-curve analysis of GEO objects", *Advances in Space Research*, Volume 58 (4), pp. 514-527, 2016.
- [4] F. Piergentili, F. Santoni, P. Seitzer, "Attitude Determination of Orbiting Objects from Lightcurve Measurements", *IEEE Transactions on Aerospace and Electronic Systems*, Volume 53 (1), pp. 81-90, 2017
- [5] M. Porfilio, F. Piergentili, F. Graziani, "The 2002 Italian optical observations of the geosynchronous region", *Advances in the Astronautical Sciences*, Volume 114 II, pp. 1237-1252, 2003.
- [6] M. Porfilio, F. Piergentili, F. Graziani, "Two-site orbit determination: The 2003 GEO observation campaign from Colleparado and Mallorca", *Advances in Space Research*, Volume 38 (9), pp. 2084-2092, 2006.
- [7] P. Morgan, *Carbon fibers and their composites*, Taylor & Francis Group, U.S.A., 2005.
- [8] J. E. Sheehan, K. W. Buesking, and B. J. Sullivan, *Carbon-carbon composite*, *Annual Review of Material Science* 24 (1994) 19-44.
- [9] G. Savage, *Carbon-Carbon Composites*, Chapman & Hall London (1993), pp. 85-113 and 227-382.
- [10] E. Fitzer, L. Manocha, *Carbon reinforcements and carbon/carbon composites*, Springer, 1998, Berlin, pp. 71-145.
- [11] A. Delfini, F. Santoni, F. Bisegna, F. Piergentili, R. Pastore, A. Vricella, M. Albano, G. Familiari, E. Battaglione, R. Matassa, M. Marchetti, "Evaluation of atomic oxygen effects on nano-coated carbon-carbon structures for re-entry applications", *Acta Astronautica*, Volume 161, pp. 276-282, 2019.
- [12] D. Micheli, A. Vricella, R. Pastore, M. Marchetti, "Synthesis and electromagnetic characterization of frequency selective radar absorbing materials using carbon nanopowders", *Carbon*, Volume 77, pp. 756-774, 2014.
- [13] D. Micheli, C. Apollo, R. Pastore, R. Bueno Morles, P. Coluzzi, M. Marchetti, "Temperature, atomic oxygen and outgassing effects on dielectric parameters and electrical properties of nanostructured composite carbon-based materials", *Acta Astronautica*, Volume 76, pp.127-135, 2012.
- [14] *NRL Arch Reflectivity Testing*, Emerson & Cuming Microwave Products.
- [15] *Technical Bulletin 101 e NRL Arch Reflectivity Test Setup*, MAST Technologies.
- [16] D. Micheli, R. Pastore, A. Vricella, A. Delfini, M. Marchetti, F. Santoni, "Electromagnetic Characterization of Materials by Vector Network Analyzer Experimental Setup" in *Spectroscopic Methods for Nanomaterials Characterization*, Elsevier, pp. 215-220, 2017.
- [17] R. Pastore, A. Delfini, D. Micheli, A. Vricella, M. Marchetti, F. Santoni, F. Piergentili, "Carbon foam electromagnetic mm-wave absorption in reverberation chamber", *Carbon*, Volume 144, pp. 63-71, 2019.