# SOLAR PROBE + MISSION: VUV RADIATION COUPLED TO HIGH TEMPERATURES ON CARBON/CARBON COMPOSITES

**J. Eck, M. Balat-Pichelin, J.-L. Sans**

*PROMES-CNRS, 7 rue du four solaire, 66120 Font-Romeu – Odeillo, France*
*Email: julien.eck@promes.cnrs.fr, marianne.balat@promes.cnrs.fr, jean-louis.sans@promes.cnrs.fr*

## ABSTRACT

For the next exploration of the sun, missions like Solar Probe+ (NASA) or Phoibos (ESA) will be launched to answer to fundamental questions on the solar corona and solar winds. The main objectives are the determination of the structure and dynamics of the magnetic fields at the source of solar winds, how is heated the solar corona and the consequent effect on the acceleration of solar winds. Such missions that will pass very close to the sun, respectively at 9.5 and 4 solar radii, need heat shield to protect the payload with the instrumentation. Carbon/carbon composites can withstand the severe environment encountered during the pass of the sun.

In order to reproduce partially these extreme conditions, high vacuum, high temperature on materials exposed to bombardment with energetic particles (hydrogen or/and helium ions) and Vacuum-Ultra-Violet (VUV) irradiation have to be implemented on ground simulation facilities. In this way, the MEDIASE facility was designed in collaboration with CNES and installed at the focus of the 1 MW solar furnace of PROMES-CNRS laboratory. Several C/C composites and one grade of polycrystalline graphite were studied. Results on the combined interaction of high temperature and VUV radiation are presented and compared to the only effect of high temperature heating on the tested materials. According to the elaboration method and the post-thermal treatment during the whole elaboration processes of the composite materials, some differences in the physico-chemical behavior of such samples are shown.

Before and after the treatment of the samples, analyses are performed using several adapted techniques such as SEM, XRD and Raman. An important effect of the VUV radiation was observed if compared to the only high temperature treatment that can affect the measurements done by the payload instrumentation.

## 1. THE SOLAR PROBE+ MISSION

The aim of Solar Probe+ (SP+) is to understand how the solar corona is heated and how the solar wind is accelerated [1]. To complete this mission, SP+ (Fig. 1) has to approach as close as 9.5 solar radii (Rs) from the Sun and has to withstand extreme environmental conditions such as high temperature, particle bombardment and Vacuum-Ultra-Violet (VUV) radiation. To protect the on-board instruments from these aggressions a Thermal Protection System (TPS) constituted of a carbon/carbon composite primary heat shield (coated or not) is considered. At closest approach, the TPS can reach temperatures up to about 1600 K according to the thermo-radiative properties of the material. Sun environment also presents energetic particles coming from the solar wind. These particles are mainly hydrogen and helium ions and can induce radiation effects on carbon/carbon composites such as physical sputtering, chemical sputtering and radiation enhanced sublimation (RES) [2, 3]. Moreover, VUV radiation coming from the chromosphere can induce additional damages on the heat shield material. In the way to determine the physico-chemical behavior of potential candidate materials designed for the TPS of Solar Probe+ the synergistic effect of high temperature, VUV radiation and particle bombardment have to be investigated.

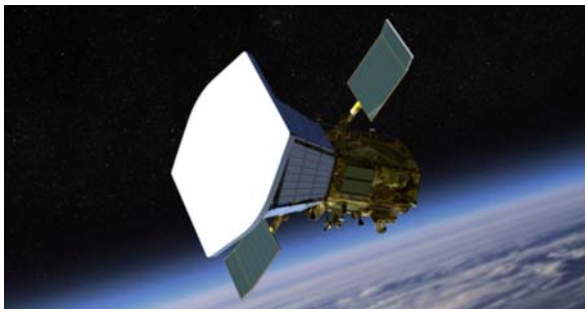

Fig. 1: Artist view of Solar Probe+
(Courtesy of NASA)

## 2. NUMERICAL STUDY

The numerical study was performed using PSIC (Plasma Surface Interaction Codes). These FORTRAN subroutines [4] implement published semi-empirical formulae for physical sputtering, chemical sputtering and RES of graphite. As chemical sputtering and RES depend on temperature the calculation codes integrate the steady state one-dimension heat diffusion equation for graphite. PSIC is a simple model and is not valid

for fully quantitative simulation of experiments. In the way to model the interaction between the solar wind and the carbon/carbon composite constituting the TPS the performed calculations took into account hydrogen and helium ions and their different energies of incidence characteristics of the slow and fast solar winds. The energies for the slow solar wind were respectively set to 2 keV ($H^+$) and 8 keV ($He^{2+}$), and 4 keV ($H^+$) and 13 keV ($He^{2+}$) for the fast solar wind. The ion fluxes also depends on the type of solar wind and were respectively set to $4.6x10^{15}$ H $m^{-2}$ $s^{-1}$ and $2.6x10^{14}$ He $m^{-2}$ $s^{-1}$ for the slow solar wind, and $1.1x10^{15}$ H $m^{-2}$ $s^{-1}$ and $7.4x10^{13}$ He $m^{-2}$ $s^{-1}$ for the fast solar wind. The proportion of hydrogen and helium does not change from one wind to another and is approximately 95 % H and 5 % He.

The physical sputtering yield was calculated with the Bohdansky formula [5] at normal incidence and the procedure specified by Yamamura [6] is used to specify the angular dependence. The two influent parameters are the mass and the energy of the incident particles (Fig. 2). Indeed, the physical sputtering yield decreases when the energy of incidence of each particle increases: from 2.2 to 1.3 % for H energy from 2 to 4 keV and from 6 to 4.5 % for He energy from 8 to 13 keV. In these energy ranges, the mass of the incident particles also induces an increase of the physical sputtering yield of near a factor of 3.

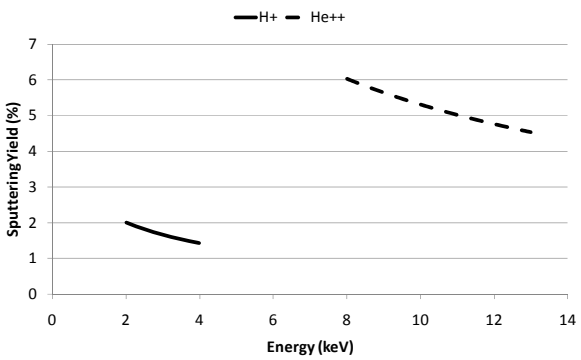


Fig. 2: Physical sputtering yield of carbon under hydrogen and helium bombardment

The chemical sputtering yield of carbon by hydrogen was calculated with the chemical erosion formula proposed by Roth [7]. From 1500 to 1800 K, a gap was noticed between the chemical sputtering yield induced by slow solar wind and the chemical sputtering yield induced by fast solar wind (Fig. 3). This phenomenon was mainly explained by the difference of incident ion fluxes and energies from one wind to another. A decrease of the chemical sputtering yield was also observed with the increase of the target temperature. Thus the calculated contribution of the chemical erosion can be neglected beyond 1800 K for fast solar wind and beyond 2000 K for slow solar wind.

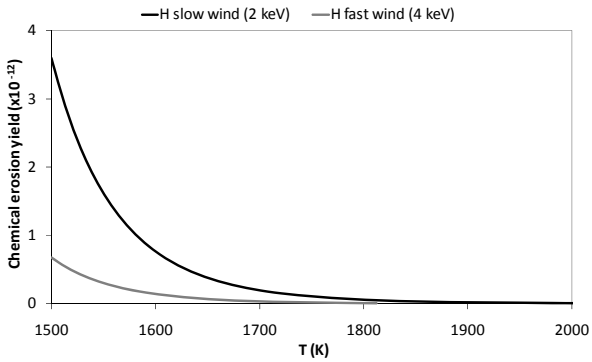


Fig. 3: Chemical sputtering yield of carbon by hydrogen at 10 Rs for slow and fast solar winds

The last phenomenon considered was the RES yield [8] which increased exponentially with temperature (Fig. 4). The mass of the incident particles is an influent parameter and lead to a RES yield due to helium about 5 times higher than the RES yield due to hydrogen on the whole range of temperature.

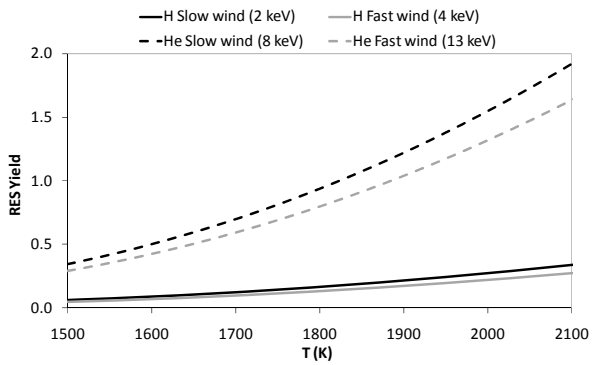


Fig. 4: RES yield of carbon by hydrogen and helium at 10 Rs for slow and fast solar winds

## 3. EXPERIMENTAL STUDY

The MEDIASE (Moyen d'Essai et de Diagnostic en Ambiance Spatiale Extrême) test facility has been designed and instrumented to reproduce the solar environment and characterize in-situ materials by analyzing their outgassing and measuring their mass loss and thermo-radiative properties. This set-up is implemented at the focus of the 1 MW solar furnace of the PROMES-CNRS laboratory (Fig. 5).

This set-up allows heating samples up to 2500 K, under high vacuum ($10^{-7}$ hPa), with a maximal ion flux of 5 keV and $10^{16}$ ions.$m^{-2}$.$s^{-1}$ and a VUV photons flux up to $10^{18}$ photons.$m^{-2}$.$s^{-1}$ for the H Lyman $\alpha$ line. This set-up is instrumented to carry out *in situ* measurements using a quartz crystal microbalance, an open source mass spectrometer, a pyro-reflectometer for true temperature measurement and a radiometer to measure the thermo-radiative properties of the materials that are conditioning the thermal equilibrium

of the heat shield – particularly the α/ε ratio of the solar absorptivity α to the total hemispherical emissivity ε.

The materials can be heated up to 2500 K in few seconds by concentrated solar radiation passing through a hemispherical silica window placed in front of the chamber under high vacuum ($10^{-6}$ hPa). On the rear face of the sample, temperature, mass loss and optical measurements can be performed. Several emplacements have been implemented around the chamber to insert the ion and VUV sources and the measuring instruments.

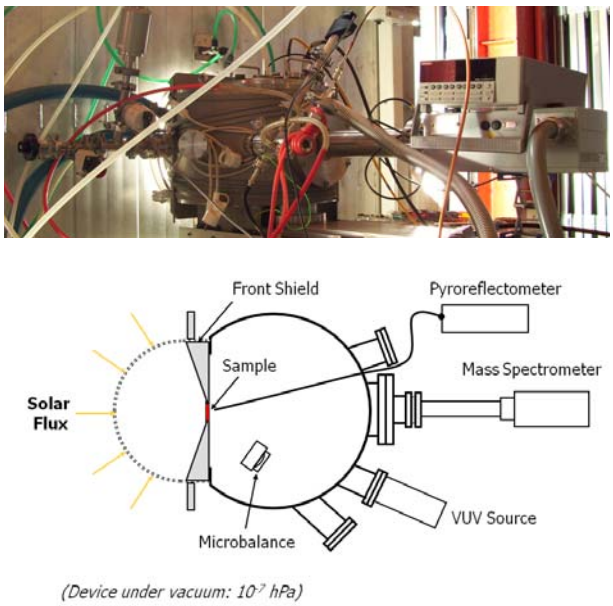


Fig. 5: Picture and sketch of the MEDIASE facility

The studied materials were successively heated at 1600, 1800 and 2100 K during 15 min for each level of temperature. They were also irradiated by VUV photons with an Omicron HIS13 source. The operation of the lamp is based on the principle of a cold cathode capillary discharge. The inlet gas used is hydrogen, in the way to obtain the H Lyman α line at 121.6 nm, characteristic of the VUV solar spectra. The photon flux reached is about 1 W $m^{-2}$, corresponding to a distance of nearly 15 Rs. The test facility allows to perform several measurements when the sample is heated and irradiated. A Leybold-Inficon IC/5 Quartz Crystal Microbalance (QCM) allows to record the mass loss rate of the samples with an accuracy of 0.2 µg $m^{-2}$ and a Hiden HAL4 EPIC300N quadrupole mass spectrometer analyzes the gaseous species emitted by the material during the experiment.

Table 1: Characteristics of the ASB and the SEPG C/C composites

| C/C composites | ASB | SEPG |
|---|---|---|
| Fiber | Ex-PAN HR | |
| Preform | 2.5 D | |
| Weave | Twill weave | Plain weave |
| Liquid consolidation | Yes | No |
| Final thermal treatment | 2470 K | |

The study focused on 2 carbon/carbon composites (ASB and SEPG) which characteristics are reported in Table 1. The ASB composite was elaborated by EADS-Astrium (France) and the SEPG composite was elaborated by Snecma Propulsion Solide (France). The 2 materials are based on ex-PAN HR fibers and a 2.5 D fibrous perform. They differ only by the weave and the liquid consolidation.

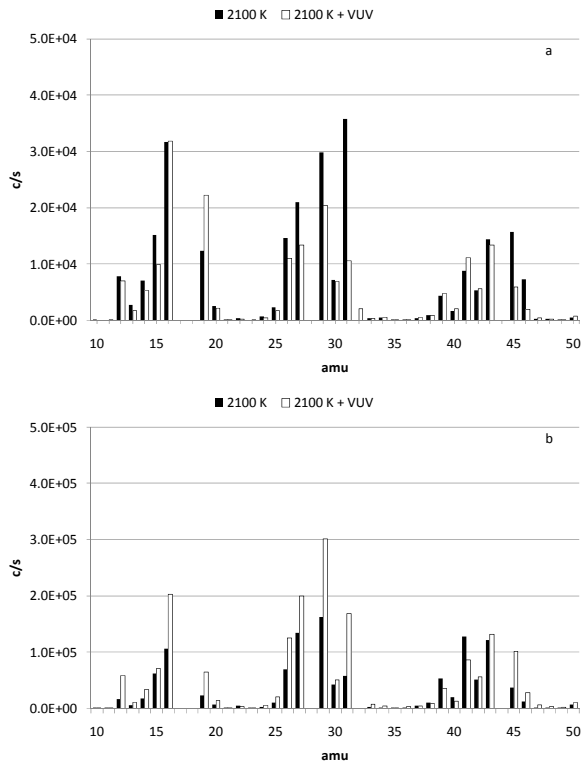


Fig. 6: Gaseous species emitted at 2100 K under high temperature with or without VUV by (a) the SEPG composite and (b) the ASB composite

The gaseous species emitted by the samples at 2100 K were monitored by mass spectroscopy. Three groups can be observed on the graphs corresponding respectively to C, $C_2$ and $C_3$ atoms and their related hydrocarbons. The SEPG composite (Fig. 6, a) emitted less gaseous species than the ASB composite (Fig. 6, b): nearly one order of magnitude whether the test conditions. When VUV radiation was added to high temperature, the 2 carbon/carbon composites

showed different behaviors. While almost no modification was recorded for the gaseous species emitted by the SEPG composite except a slight decrease of the $C_2$ group, the ASB composite presented an opposite behavior with a global increase of the gaseous species emitted.

In the same way, the mass loss rate of the 2 materials presented different tendencies, as reported in Table 2. The SEPG composite lost less mass than the ASB composite and only at 1600 K while the ASB show mass loss on the whole temperature range. However, none of the values overcome the NASA mass loss requirement of 0.44 mg $m^{-2}$ $s^{-1}$. Here again, when VUV radiation was added to high temperature, the behavior of the 2 carbon/carbon composites became different. At 1600 K, the recorded mass loss for SEPG decreased whereas it increased for ASB. At 1800 K, the ASB mass loss decreased and became null at 2100 K.

Table 2: Mass loss rate of the ASB and SEPG composites at high temperature with or without VUV

| **Δm (mg $m^{-2}$ $s^{-1}$)** | **ASB** | | **SEPG** | |
|---|---|---|---|---|
| T (K) | HT | HT+VUV | HT | HT+VUV |
| 1600 | 0.09 | 0.35 | 0.12 | 0.03 |
| 1800 | 0.19 | 0.17 | 0 | 0 |
| 2100 | 0.07 | 0 | 0 | 0 |

The morphology of the samples were also studied using secondary electron microscopy (SEM) but except a cleaning of the surface, no significant modifications is noticed on the surface of the ASB and SEPG carbon/carbon composites as can be seen on Fig. 7.

The evolution of the material microstructure was studied by micro Raman spectroscopy using a 647.1 nm argon-krypton laser. The G band located at 1580 $cm^{-1}$ is characteristic of the graphite microstructure while the D band at 1350 $cm^{-1}$ implies structural disorder. The SEPG composite presents almost no microstructural modification, whatever the test parameters. On the contrary, microstructural modifications can be seen for the ASB composite (Fig. 8). At high temperature, there is almost no variation of the defect concentration but the material is further crystallized. Under combined high temperature and VUV radiation, both defect concentration and crystallinity are increased.

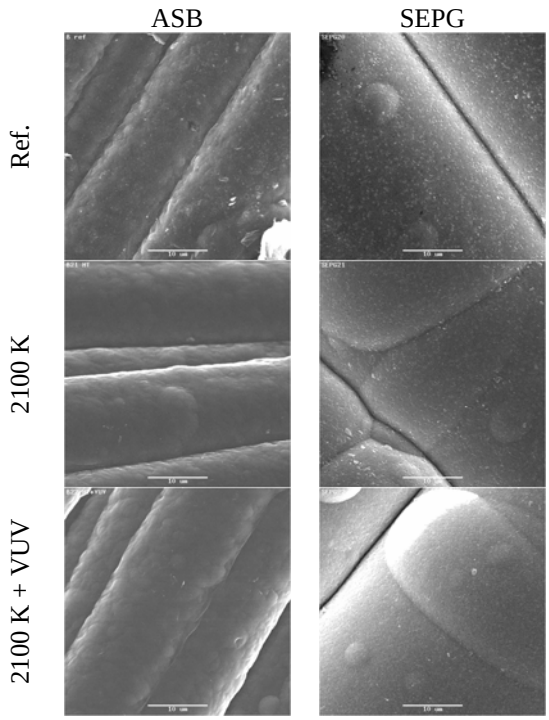


Fig. 7: SEM micrographs of the ASB and SEPG composites. The scale bar is 10 µm

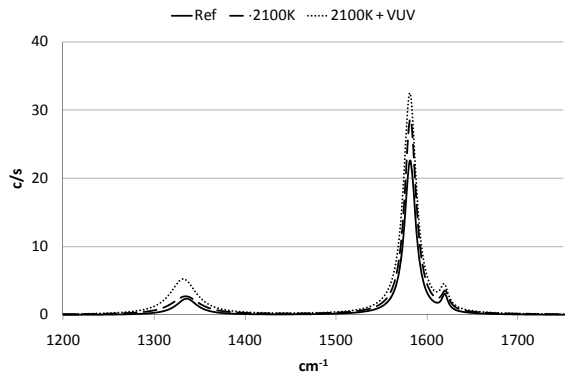


Fig. 8: Raman spectra of the ASB composite

Finally, the thermo-radiative properties of the carbon/carbon composites were investigated using a direct method [9]. The ratio of the solar absorptivity α (0.6 - 2.8 µm) to the total hemispherical emissivity ε (0.6 – 40 µm) is a critical point to determine the thermal equilibrium of the heat shield of Solar Probe+ at perihelion (9.5 Rs). The results are acquired using a radiometer and a three-mirror goniometer added to the MEDIASE facility.

Results show that the ASB and SEPG carbon/carbon composites have similar thermo-radiative properties, with a α/ε ratio from 1.2 to 1.1 on the whole temperature range (Fig. 9). Moreover, VUV radiation did not change the thermo-radiative properties of the materials.

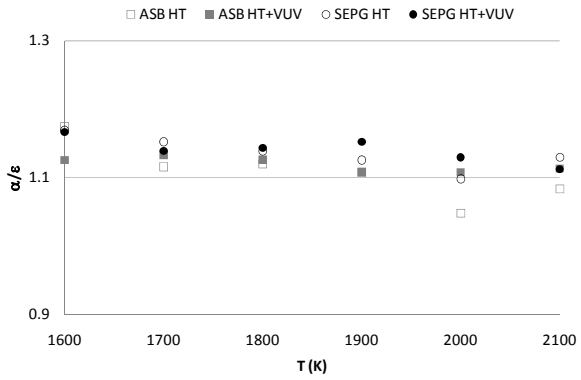


Fig. 9: Thermo-radiative properties of the ASB and SEPG composites at high temperature

## 4. CONCLUSION

The aim of this study was to describe the physico-chemical behavior of two potential candidate materials for the thermal protection system of Solar Probe+ under high temperature and under high temperature with VUV radiation. From a numerical point of view, only the physical sputtering and the radiation enhanced sublimation of graphite could induce significant degradation of the heat shield material whereas chemical sputtering could be neglected at the considered temperature level (from 1600 to 2100 K).The experimental study showed that the elaboration process of the composite had an impact on the physico-chemical behavior of the material. Indeed, even if they both underwent a final thermal treatment at a higher temperature than the ones applied during the experiment, differences between the behavior of ASB and SEPG composites were observed. The SEPG carbon/carbon composite emitted less gaseous species and presented a very low mass loss. Moreover, its microstructure and its thermo-radiative properties were not altered by high temperature nor high temperature with VUV radiation. On the contrary, ASB shown a different behavior. Its emission of gaseous species was more important, about one order of magnitude, and was further increased by the addition of the VUV radiation. Its mass loss was also more important and was first increased by VUV radiation at 1600 K and then decreased at 1800 K and 2100 K. Concerning the microstructural modifications, they were significant both at high temperature and when VUV radiation was added. A great increase of the structural disorder was noticed. However these alterations had no impact on the thermo-radiative properties of the material.

The perspective of this study is to add ion bombardment to high temperature and VUV radiation in the way to reproduce accurately the extreme environmental conditions that Solar Probe+ will encounter at perihelion during the mission.

## 5. REFERENCES

1. Solar Probe Plus, *Report of the Science and Technology Definition Team*, July 2008, NASA/TM-2008-214161.

2. Paulmier T., Balat-Pichelin M., Le Quéau D., Berjoan R., Robert J.F., Physico-chemical behavior of carbon materials under high temperature and ion irradiation, *Appl. Surf. Sci.* 180, 227-245, 2001.

3. Paulmier T., Balat-Pichelin M., Le Quéau D., Structural modifications of carbon-carbon composites under high temperature and ion irradiation, *Appl. Surf. Sci.* 243, 376-393, 2005.

4. Warrier M., Schneider R., Bonnin X., Subroutines for some plasma surface interaction processes: physical sputtering, chemical erosion, radiation enhanced sublimation, backscattering and thermal evaporation, *Comput. Phys. Comm.* 160, 46-68, 2004.

5. Bohdansky J., A universal relation for the sputtering yield of monatomic solids at normal ion incidence, *Nucl. Instrum. & Methods Phys. Res. Sect. B* 2, 587-591, 1984.

6. Eckstein W., Computer simulation of ion surface collisions with special emphasis to the work of Yamamura, *Nucl. Instrum. & Methods Phys. Res. Sect. B*, 232, 108-116, 2005.

7. Roth J., Chemical erosion of carbon-based materials in fusion devices, *J. Nucl. Mat.* 266-269, 51-7, 1999.

8. Roth J, Vietzke E, Haasz AA, Erosion of graphite, *Suppl Nucl Fusion* 1, 63-78, 1991.

9. Balat-Pichelin M., Robert J.F., Sans J.L., Emissivity measurements on carbon–carbon composites at high temperature under high vacuum, *Appl. Surf. Sci.*, 253, 778-783, 2006.