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Microstructure and gas-surface interaction studies of a low-density carbon-bonded carbon fiber composite in atmospheric entry plasmas

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ABSTRACT

Carbon-bonded carbon fiber (CBCF) composites are a cost-effective solution for the production of low-density carbon-phenolic Thermal Protection Systems (TPS). This new TPS for spacecraft requires new experimental data for model development and validation. Ablation experiments of a CBCF composite were carried out in an inductively-coupled plasma generator to assess the performance in high-enthalpy flows. Surface temperatures up to 2900 K led to strong surface ablation and test samples of hemispherical shape responded with constant surface temperatures and recession rates. Cylindrical samples experienced a continuous surface temperature increase. Emission spectra of the cyano radical CN were indicative of a 4–5 mm reactive boundary layer. Deviation from thermal equilibrium was found by comparison to simulated spectra. Micrographs revealed an oxidation zone in the order of 0.2 mm at the surface, suggesting a gas phase diffusion controlled ablation regime. Strong corrosion of the fibers in nitrogen plasma is attributed to wall nitridation.

1. Introduction

Ablative Thermal Protection Systems (TPS) are currently the only solution for future space exploration missions that aim at returning samples from Mars and asteroids at very high reentry speeds, in order to protect the spacecraft from severe heating. To achieve high performance characteristics of the ablative Thermal Protection Material (TPM) engineers of planetary probes and space vehicles make use of a wide range of composites [1,2]. Ablative heat shields are generally composed of a rigid precursor and a filling matrix, to serve as a pyrolyzing, ablating, and insulating material at low weight with reasonable mechanical properties. In this way, they are able to dissipate high heat fluxes through chemical and physical decomposition, transforming the thermal energy into mass loss and surface recession, whilst the remaining solid material insulates the vehicle substructure [3,4]. A current example of a new family of porous Lightweight Ceramic Ablators (LCA) is made of a carbon fiber preform impregnated with phenolic resin such as PICA developed by NASA [5,6], and European ablator Asterm, developed by Airbus DS [7]. Besides, several research groups started on an academic level to manufacture and characterize new lightweight ablators [8–10].

Selection and thickness definition of the TPM are the two key performance parameters in TPS design. Prediction inaccuracies can be fatal for the crew or the success of robotic missions. Consequently, ground testing in plasma wind-tunnels becomes a fundamental requirement for TPM qualification, validation of material response codes, and TPS design. With the work presented in this paper, we want to contribute with new experimental data to the ongoing efforts of improving the heat shield reliability by reducing design uncertainties and developing new thermo-chemical ablation models. The non-pyrolyzing material is very similar to the rigid precursor for Asterm and PICA, and serves as an ideal candidate to decouple the physico-chemical phenomena of heterogeneous pyrolysis gas chemistry and carbon fiber/char ablation for fundamental ablation experiments.

Despite the extensive use of a broad variety of ablators during past missions, and efforts in material modeling [11,12], prediction of the heat flux to the sub-surface of a spacecraft remains a critical problem. Since the start of space flight and the development of charring ablators, graphite was the material of choice for fundamental ground tests of carbon ablation, leaving the complex pyrolysis chemistry and boundary layer contamination of outgassing ablators aside. Many authors have addressed graphite ablation in dependence of surface temperature, and gas pressure and classified it into reaction controlled, diffusion controlled, and sublimation controlled ablation regimes [13,14]. Following the revolution of new carbon-composites in many fields, the oxidation properties

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of carbon–carbon materials at high temperatures are demanded for a wide range of applications and have been subject to a large variety of investigations [15–18]. A major outcome from those studies is that the ablation mechanism, and hence the reactivity, strongly depends on the type and density of carbon fibers (themselves relying on the precursor type, and on the processing & graphitization cycles [19]), their connecting interface and defects on the fiber surface. New multi-scale material response models are proposed [20–22] in order to take into account the porous micro-structure of the new class of materials. Volume-averaging of this microscopic scale model helped analyzing and better understand the oxidation behavior of carbon fibers, embedded in the charred phenolic-polymer matrix.

The prediction tools for ablative TPS design are still inherited from the Apollo program, and were originally developed for different types of materials. The control of the material response of the new generation, low density carbon composite ablators in environments similar to real reentry conditions requires comprehensive experimental data in well-characterized high-enthalpy facilities. Most ground-based investigations on TPM have been carried out in arc-jet facilities, which offer high-enthalpy, supersonic plasma flows in order to reproduce stagnation pressure and peak heating on the material surface. This strategy allows qualification of a specific material in a confined test environment, but extrapolation to flight values is difficult and material response models were designed to match ground test data [23]. A detailed description of the plasma flow and the reactive/diffusive environment in the boundary layer is often poorly addressed. The subsonic 1.2 MW Inductively Coupled Plasma (ICP) torch of the Plasmatron facility at the von Karman Institute (VKI) is able to reproduce the aero-thermodynamic environment of atmospheric entry in the boundary layer of a test object for a wide range of pressures and heat fluxes [24,25]. An extensive numerical rebuilding procedure offers a detailed characterization of the boundary layer and extrapolation of ground-test data to real re-entry flight conditions. Hence, we believe that our experimental approach, consisting of a comprehensive experimental setup and the non-pyrolyzing carbon fiber precursor material along with a microscale analysis, will provide a unique class of complementary data.

Cylindrical and hemispherical samples were tested at 1.5–20 kPa and surface temperatures of 1900–2800 K. The main focus of the study is the recession of the material in a well characterized test gas environment, but the scope of our observations covers three main aspects:

1. **Experimental methods**: new approaches to asses the performance of various aerospace applied composites in service including in situ ablation observation and microstructural characterization.
2. **Test material**: how does the porous carbon-bonded carbon composite, similar to the precursor of new generation ablators, perform in different pressure and heat flux conditions in the absence of pyrolysis.
3. **Gas-surface interaction**: steady-state, well-characterized test conditions, providing high-quality data for new approaches to prediction of the physical and chemical behavior in atmospheric entry plasmas.

2. Experimental techniques

2.1. Test material

The bulk material of highly porous carbon-bonded carbon fiber preform (CBCF) was provided by Mersen Scotland Holytown Ltd. (CALCARB® CBCF 18-2000 [26]) and consists of a short fiber insulation originating from rayon. The chopped, discontinuous virgin carbon fibers are interconnected in a matrix produced by the carbonization of phenolic resin. During this process the fibers become oriented and the microstructure and properties are anisotropic. The material is then vacuum-treated at temperatures above 2300 K to ensure its temperature stability and the absence of outgassing. According to the supplier, the material contains no more than 500 ppm of impurities. Main problems related to the use of CBCF are mechanical erosion and the poor oxidation resistance typical of carbon-based composites [27]. The material was machined in-house to cylindrical (CY) and hemispherical (HS) test samples, both of radius 25 mm with 45 mm (cylindrical) and 50 mm (hemispherical) in length. The samples have an initial density of about 180 kg/m^2 with an initial porosity of 90%. Physical and thermal properties of CBCF 18-2000 are listed in Table 1. This non-pyrolyzing material is very similar to the rigid precursors of Asterm and PICA, with the latter of the two using Fiberform® [28] as preform prior to impregnation with phenolic resin.

Test sample shape and size have a strong influence on the boundary layer chemistry, which is driven to a great extent by the effective radius of the front surface. Depending on the chemistry relaxation time, and the time scale at which the flow is able to reside in the boundary layer of the test sample, the chemistry can change from equilibrium to frozen conditions. This will be further explained in Section 2.2.2.

A Kern EW150-3M balance (1 mg precision) and a 0.1 mm precise caliper gauge were used for pre- and post-test evaluation of recession (beside a high-speed camera (HSC) imaging technique) and mass loss of the samples. The sample was attached to a sample holder, with its stagnation point located 445 mm downstream of the plasma jet exit, outside of the plasma flow prior to exposure. After reaching the desired test condition and calibration of the plasma flow, the sample was injected using a pneumatic mechanism. Images of pre- and post-test samples are presented in Section 3.1.

2.2. Plasma wind tunnel overview

The VKI Plasmatron facility has been used for all the experiments for the reproduction of the aero-thermodynamic environment of re-entry plasma flows, creating a high-enthalpy, highly dissociated subsonic gas flow. First ablation experiments on the

Table 1
Physical properties of CBCF 18-2000 [26].

Bulk density (kg/m^3)	Compressive strength (MPa)		Flexural strength (MPa)		Spec. surface area (m^2/g)	Thermal conductivity (W/m K)			
180 ± 30	1.1[a]	0.76[b]	1.03[a]	0.15[b]	18		Vac	N_2	Ar
						500 °C	0.26	0.48	0.36
						1000 °C	0.41	0.72	0.54
						2000 °C	1.00	1.47	1.16

[a] Parallel to fiber orientation (xy).
[b] Perpendicular to fiber (z).