International Journal of Heat and Mass Transfer 100 (2016) 810–824

Contents lists available at ScienceDirect

International Journal of Heat and Mass Transfer

journal homepage: www.elsevier.com/locate/ijhmt

Experimental investigation of ablation and pyrolysis processes of carbon-phenolic ablators in atmospheric entry plasmas

Bernd Helber [a,b,*], Alessandro Turchi [a], James B. Scoggins [a], Annick Hubin [b], Thierry E. Magin [a]

[a] von Karman Institute for Fluid Dynamics, Chaussée de Waterloo 72, B-1640 Rhode-Saint-Genèse, Belgium
[b] Vrije Universiteit Brussel, Pleinlaan 2, B-1050 Brussel, Belgium

ARTICLE INFO

Article history:
Received 22 September 2015
Received in revised form 10 December 2015
Accepted 20 April 2016

Keywords:
Charring ablator
Pyrolysis blowing
Surface ablation
Emission spectroscopy
Atmospheric entry

ABSTRACT

We study the ablation and transient pyrolysis outgassing of the carbon-phenolic ablators AQ61 and Asterm in air and nitrogen plasmas. We investigate their resistance to high heating conditions, and characterize gas-surface interaction phenomena, including the interaction of the pyrolysis gases with the hot plasma flow. The experiments were carried out in the Plasmatron facility of the von Karman Institute for Fluid Dynamics. The aero-thermodynamic environment of atmospheric entry in the boundary layer of a test object was selected with surface temperatures between 1900 K and 2800 K, and test chamber pressures of 15 hPa, 100 hPa, and 200 hPa.

Those conditions led to recession rates between 39 μm/s and 83 μm/s in air plasmas. Micrographs revealed oxidation of the char layer and carbon fibers. Carbon deposition in the form of soot was observed on samples tested in nitrogen, contrary to air ablation where charred resin was not found at the surface.

We propose an approach to estimate the temporally resolved pyrolysis outgassing rate, based on the emission signature of pyrolysis products and the volume change of the sample. The temporal recession rate was obtained from high-speed camera imaging. This enabled evaluation of the surface recession as a function of the pyrolysis outgassing rate, which was then compared to numerical estimates predicted by thermochemical equilibrium tables.

The thermochemical equilibrium model generally underpredicted experimental recession rates, particularly at low pressure (15 hPa). Stronger mechanical failure of the material was ruled out as experiments at the same test conditions in nitrogen plasmas did not show any significant recession. Micrographs did not indicate internal oxidation of the material, neither was spallation observed during the low pressure experiments.

1. Introduction

On 6 August 2012, NASA's Mars Science Laboratory (MSL) mission successfully landed a rover on the Martian surface including an automated sample collection system for chemistry and mineralogy analysis. The next steps will be to identify, develop, and qualify required technologies for returning Martian and other asteroid samples safely to Earth.

Such sample return missions at very high re-entry speeds will use ablative materials for the Thermal Protection System (TPS), shielding the spacecraft from the severe heating during the atmospheric entry. The ablative Thermal Protection Material (TPM) is 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. During atmospheric entry, part of the heat flux is transferred inside the heat shield, and the virgin material is transformed following pyrolysis and ablation. Pyrolysis progressively carbonizes the phenolic resin into a low density, porous char, losing around 50% of its mass producing pyrolysis gases by vaporization. The pyrolysis gases are convected out of the material and exhaust into the boundary layer, providing a further barrier for the heat exchange by blowing and undergo additional chemical reactions. Ablation of the char layer, composed of the carbonized resin and the remaining carbon fibers, is then promoted by heterogeneous chemical reactions, phase change and mechanical erosion, altogether leading to recession of the material [1–3].

A new class of lightweight carbon-phenolic composites is being developed since the last decade, specifically designed for the high heating rates of planetary missions. A current example is made of a

---

* Corresponding author at: von Karman Institute for Fluid Dynamics, Chaussée de Waterloo 72, B-1640 Rhode-Saint-Genèse, Belgium. Tel.: +32 (0)2 359 96 27.
E-mail address: helber@vki.ac.be (B. Helber).

http://dx.doi.org/10.1016/j.ijheatmasstransfer.2016.04.072
0017-9310/

porous carbon fiber substrate impregnated with phenolic resin such as PICA (phenolic-impregnated carbon ablator) developed by NASA [4,5], and European ablators Asterm and AQ61, developed by Airbus DS [6]. Most recent examples for the successful performance of PICA are the Stardust [7], and MSL [8,9] missions. A similar ablator, PICA-X, is used for the Dragon spacecraft, designed by SpaceX for crew and cargo service of the ISS.

Selection and thickness definition of the TPM are the two key performance parameters in TPS design, and rely on predictions of the heat flux to the bondline of the spacecraft. But despite the advancements made since the Apollo missions to the moon, heat flux prediction remains an imperfect science and engineers resort to safety factors to determine the TPS thickness. This goes at the expense of embarked payload, hampering sample return missions [10]. Combined theoretical, numerical, and experimental research is required to improve our understanding of the complex gas/surface interaction and ablator material response in atmospheric entry flows. Ground testing in plasma wind-tunnels is currently the only affordable possibility for both material qualification and validation of material response codes. The goal of this work is to contribute to the ongoing efforts of improving the heat shield reliability, reducing design uncertainties and developing new thermo-chemical ablation models with new experimental data on porous, low-density ablators.

Most ground-based investigations of TPM have been carried out in arc-jet facilities [5,11–16], which offer high-enthalpy, supersonic plasma flows to reproduce stagnation pressure and peak heating on the material surface. This strategy allows qualification of a specific material in a confined test environment, and the design of material response models that match ground test data [17]. The averaged experimental data, usually recession, mass loss, and temperatures, are used to extract thermophysical properties of the heat shield material [14].

The total mass loss of charring ablators is composed of simultaneous char layer ablation and pyrolysis of the internal resin binder (Fig. 1). But transient phenomena, such as the rapidly changing blowing rates caused by strong internal pyrolysis, are usually not captured. Especially during start of the experiment, the strong temperature rise causes almost instant vaporization of the phenolic resin near the surface. For example, the time for PICA to reach steady-state ablation in arc-jet tests is on the order of 10 s [16]. During steady-state ablation, the speed at which the surface recesses equals the speed of the pyrolysis propagation towards the virgin material ($\dot{s}_c = \dot{s}_v$). Recent material response simulations of porous ablators focused on the transient behavior of the pyrolysis gas flow. Those studies demonstrated the strong influence of the sample geometry on the strength and direction of pyrolysis outgassing [18] as well as the importance to accurately model the pyrolysis gas flow for a correct temperature prediction [19]. In addition, state-of-the-art numerical models treat the pyrolysis gas mixture as an average of decomposition products [20,21]. New high fidelity material response models are proposed to take into account the porous micro-structure of the new class of materials [22,23] as well as a non-constant elemental composition for the pyrolysis gas mixture [24,25].

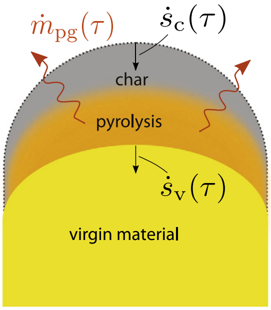

Fig. 1. Simplified schematic of mass losses on pyrolyzing ablator: Transient pyrolysis gas mass loss ($\dot{m}_{pg}$) inside the material leading to consumption of virgin material ($\dot{s}_v$), and char layer removal by recession ($\dot{s}_c$).

The focus of our study will be the identification of the transient pyrolysis gas blowing rates for evaluation of the material recession in different air and nitrogen plasma environments. 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 [26,27]. In a previous article we studied a porous, non-pyrolyzing carbon-bonded carbon preform [28], similar to the precursor of carbon-phenolic ablators such as Asterm. With this work, we extend our analysis towards phenolic-impregnated carbon ablators that may serve as TPM for future missions.

In particular, this work provides information on the following aspects:

1. Visual inspection including microscale analysis to identify ablation at the fiber scale (Section 3.1).
2. Thermal response of the material and ablation rates in air (Sections 3.2 and 3.3).
3. Tracing of pyrolysis products in the boundary layer (Section 3.4):
4. Suggestion of an experimental approach to track the transient pyrolysis gas blowing rate, enabling comparison of ablation rates with equilibrium thermochemistry (B'-tables) (Section 4).

2. Ablative materials and experimental methods

The experimental and numerical tools for this work are reviewed in this section. It includes presentation of the Asterm and AQ61 material samples, the Plasmatron facility together with experimental and numerical procedures to characterize the plasma flow field, as well as the experimental setup with description of the emission spectroscopy arrangement.

2.1. Ablative test samples

We tested two different carbon composites (Asterm and AQ61) made of short carbon fibers impregnated with phenolic resin as described below. Their geometry was a 25 mm radius hemisphere with a 25 mm (Asterm) or 20 mm (AQ61) long cylinder. In the subsonic plasma flow of the VKI Plasmatron, hemispherical ablative samples responded with a stable axial recession of the hemisphere, with low sidewall ablation [29]. This allows for constant boundary conditions throughout the whole experiment.

Asterm is a low-density ablator, similar to NASA's PICA [4], developed by Airbus DS for future high-speed (re-) entry missions. It is manufactured by impregnating a rigid graphite felt preform with phenolic resin, followed by a polymerization process and final machining [6]. This approach significantly reduces the manufacturing effort and allows for large scale production over a large range of final material densities, from 240 kg/m^3 to 550 kg/m^3. The expanded structure of impregnated resin gives the material a low density and high porosity. We already presented other Plasmatron experiments of a similar non-pyrolyzing carbon fiber preform in another reference [28]. This carbon-bonded carbon fiber (CBCF) preform is made of short cut carbon fibers, interconnected in a fully carbonized matrix [30].

AQ61 is a generic ablative material previously developed by Airbus DS, representing a low density carbon-phenolic material. In contrast to Asterm, AQ61 is not made up of one single piece of carbon