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Experimental measurements of the permeability of fibrous carbon at high-temperature

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ABSTRACT

A series of experiments was performed to obtain permeability data on FiberForm®, a commercial carbon preform used for manufacturing thermal protection systems. A porous sample was placed in a quartz flow-tube heated by an isothermal furnace. The setup was instrumented to measure mass flow through and pressure drop across the sample. The intrinsic permeability and the Klinkenberg correction, which accounts for rarefied effects, were computed from the experimental data. The role of the gas temperature and pressure on the effective permeability is shown, and it is demonstrated that with proper data reduction, the intrinsic permeability is strictly a function of the micro-structure of the material. A function for the effective permeability of FiberForm, dependent on temperature, pressure, pore geometry, and type of gas is proposed. The intrinsic permeability was evaluated at $K_0 = 5.57 \times 10^{-11}$ m^2, with a Klinkenberg parameter of $8c/d_p = 2.51 \times 10^5$ m^{-1} and a reference porosity of $\phi^i = 0.87$.

1. Introduction

The entry process into a planetary atmosphere requires spacecraft to be equipped with a thermal protection system (TPS). The TPS protects the spacecraft from the high enthalpy and thermochemical conditions of entry, during which the hypersonic flow surrounding the vehicle generates strong aerothermal heating. An ablator is usually used as a TPS material for the harshest entry conditions due to the chemical and physical phenomena that take place when high heat fluxes are experienced. The ablator materials significantly reduce the heat to the inner parts of the vehicle, protecting the payload [1]. In recent years the focus has veered toward a new class of low-density carbon/resin ablators, the most successful of which is NASA's own phenolic-impregnated carbon ablator (PICA) [2], used in Earth return and Mars exploration missions [3,4].

PICA uses FiberForm® (Fiber Materials, Inc.) [5], a rigid carbon fiber composite, as a substrate. As shown by the scanning electron micrograph in Fig. 1, its micro-structure is characterized by thin carbon fibers (≈10 μm in diameter) and pores of ≈50 μm in diameter [6]. The pores occupy nearly a 90% fraction of the volume of the material, providing it with excellent insulation properties.

Because of their high porosity, gases can easily flow within the ablative materials. For example, pyrolysis gases produced by decomposition of the phenolic resin travels through the charred structure – potentially reacting with the fibers – before exiting the material. Likewise, reactants from the boundary layer can enter the material microstructure and flow within the pores. This gas transport has a significant effect on the overall material response [7–9].

The flow behavior through a porous structure is characterized by the permeability, as it dominates the momentum transport within the medium. Permeability is therefore a key material property when modeling porous media flow.

When the mean free path λ of the gas molecules approaches the dimensions of the material pores, the gas flow within the material is considered transitional between the continuum and Knudsen regimes. In this regime, slip effects become important.

A method for measuring the permeability of porous refractory insulators was proposed by Marschall and Milos [10,11] and applied to various materials, such as silica-based tiles, PICA

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Nomenclature

Symbols

$\mathcal{R}$	universal gas constant [J/(K mol)]
δf	frequency resolution [s^{-1}]
ΔP	pressure difference [Pa]
$\dot{m}$	mass flow rate [kg/s]
A	area of the flow-tube [m^2]
b	permeability slip parameter [Pa]
c	proportionality constant
d	diameter [m]
F	resistive force [N]
K	permeability [m^2]
L	length of the sample [m]
M	molar mass [kg/mol]
P	pressure [Pa]
T	temperature [K]
t	time [s]
u	gas velocity [m/s]
x	spatial coordinates [m]

Greek symbols

λ	mean free path [m]
μ	viscosity [kg/(m s)]
ϕ	porosity [m^3/m^3]
ρ	density [kg/m^3]

Superscripts

$\dagger$	values scaled at $\phi = 0.87$
$*$	values scaled at $T = 298$ K

Subscripts

0	intrinsic
1	port P1
2	port P2
avg	average across sample
eff	effective
f	furnace
fib	fiber
p	pore
s	surface

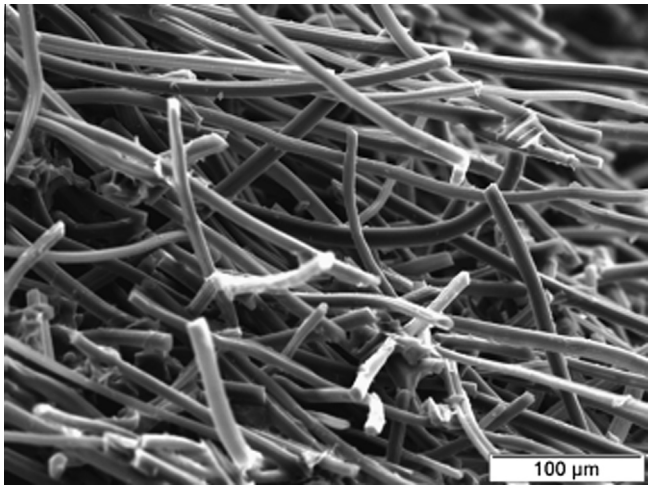

Fig. 1. Scanning electron micrograph of FiberForm.

(in virgin and charred from), ceramics, and to a lesser extent, FiberForm. Data on FiberForm in Ref. [10] were obtained up to 300 K on an older, less dense version of the material with large observed variabilities in the samples.

The experiments documented in the current work provide an updated set of FiberForm permeability values at temperatures ranging from 298 to 1500 K, in inert atmosphere. The data generated also constitute a baseline for the numerical rebuilding of effective reactivity data from experiments on the high-temperature decomposition of FiberForm [12].

2. Experiment

A high-temperature flow-tube setup (Fig. 2) was assembled to perform gas/material interaction experiments on porous samples. The system consisted of a 129.5 cm long, 22 mm inner diameter quartz tube positioned inside of an open-ended furnace providing temperatures up to 1675 K by means of a radiative ceramic element. The cylindrical plug samples (FiberForm) were inserted in the tube by interference fitting, and positioned in the center of the furnace, using a plastic dowel rod. As discussed in the related literature [10,11], the axial geometry of the porous material plays a major role in the permeability.

Because of its manufacturing method, FiberForm is an orthotropic material. More specifically, it is transverse isotropic, since most of the fibers are oriented within ±15° of the compression plane. The direction perpendicular to this plane is defined as "Through-Thickness" (TT) and that parallel as "In-Plane" (IP). The bulk of the experiments described here were performed on samples machined with a TT orientation, in which the carbon fibers are preferentially aligned perpendicular to the gas flow direction. One experiment was also performed with a sample oriented in the IP direction. A dedicated mass flow controller (Aalborg Model: UFC 8160) calibrated to nitrogen trifluoride was controlled by a Tylan RO-28 Readout/Control Box to feed the argon gas at fixed flow rates ranging between 10 and 100 sccm. The system was evacuated by means of an Alcatel R301B Roots pump using Fomblin® oil and backed by an Alcatel BF ADP 81 dry pump. The pumping manifold was outfitted with a copper mesh to collect particulates that might be emitted during the experiment. The outlet of the flow-tube was connected to the vacuum system through a manual bellows-angle valve, fully opened during the experiments.

The main tube was equipped with both an upstream (P1) and downstream (P2) port from the furnace, which were connected to a manifold of calibrated differential pressure transducers measuring the pressure loss ($P_1 - P_2$) across the sample. A separate set of pressure gauges were also used to monitor absolute pressure conditions.

A valving manifold was used to control gas flows and normalize pressure in the system when starting experiment operations. One set of these valves was used as a by-pass to prevent the formation of strong pressure gradients across the sample during evacuation or venting operations that could potentially move the sample from the desired initial position. Thermocouple (TC) sensors were installed at different strategic positions along the tube as depicted in Fig. 2. Two Type-K thermocouples were used to measure the temperature T_1 and T_2 at the pressure ports P1 and P2, respectively, and two other Type-K TCs monitored the temperature T_{in} and T_{out} at the inlet and outlet of the furnace. A Type-S and