International Journal of Heat and Mass Transfer 108 (2017) 801–811

Contents lists available at ScienceDirect

International Journal of Heat and Mass Transfer

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

Micro-tomography based analysis of thermal conductivity, diffusivity and oxidation behavior of rigid and flexible fibrous insulators

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ARTICLE INFO

Article history:
Received 9 September 2016
Received in revised form 18 November 2016
Accepted 15 December 2016

Keywords:
A. Carbon fibers
B. Porosity
B. Thermal properties
B. Oxidation

ABSTRACT

Material properties and oxidation behavior of low-density felts used as substrates for conformal carbon/phenolic ablators were compared with those of a rigid carbon fiber preform used to manufacture heritage lightweight ablators. Synchrotron X-ray micro-tomography measurements were performed to characterize the materials' microstructure at the scale of the fibers. Using the tomography voxels as computational grids, tortuosity in the continuum regime, and room temperature conductivity were computed. Micro-scale simulations of the oxidation of carbon fibers were carried out using a random walk model for oxygen diffusion and a sticking probability law to model surface reactions. The study shows that, due to a higher porosity and lower connectivity, the felt materials have lower thermal conductivity but a faster recession rate than that of the rigid preform. Challenges associated with computations based on micro-tomography are also discussed.

1. Introduction

The successful atmospheric entries of Stardust, Mars Science Laboratory (MSL) and the recent flights of the SpaceX Dragon capsule have demonstrated a substantial maturity and performance of lightweight carbon/phenolic thermal protection systems (TPS). This class of lightweight ablators, with densities ($\sim$0.3 g/cm^3) much lower than fully dense carbon/phenolic ($\sim$1.5 g/cm^3), has proven capable of handling the aerothermal heating over a wide range of entry speeds and in different atmospheric environments.

In order to prepare for future exploration missions and to meet the more demanding thermal protection requirements of anticipated atmospheric entries, NASA is improving material response models for low-density carbon/phenolic ablators and developing new material technologies that enable better performance and more effective design. Examples of these technologies are conformal and flexible carbon/phenolic composites [1,2]. Their basic architecture is obtained infusing a phenolic resin into an insulating fibrous substrate, following the flight-proven state-of-the-art architecture of the Phenolic Impregnated Carbon Ablator (PICA) [3]. However, unlike PICA, which is based on a rigid preform, new materials with high application potential adopt flexible felts as the material substrate [1,2]. Felt substrates suitable for flexible and conformal ablators are based on ceramic, polymers or carbon fibers. An example of such technology is Conformal-PICA (also referred to as C-PICA) [1,2], where a rayon-based carbon felt is infused with phenolic. These new materials have been successfully tested in arcjet environments at heat fluxes ranging from 50 to 385 W/cm^2 [1,2].

Felt-based ablators have several advantages over classical rigid TPS materials. Most importantly, they mitigate the limited strain response of large rigid substrates. For example, the ceramic material used for the Space Shuttle [4] and the heritage PICA of the large MSL heat shield [5], due to their brittleness, needed to be manufactured as tiles of limited dimensions. Enabling manufacturing in larger pieces, felt based substrates reduce the number of independent parts mitigating the need of gap fillers. They also offer improved robustness in absorbing loads and deflections, and they allow shaping the substrate around complex geometries with the possibility of maintaining a uniform and low thermal conductivity in

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http://dx.doi.org/10.1016/j.ijheatmasstransfer.2016.12.048
0017-9310/

presence of curved surfaces [1]. In contrast, felts have drawbacks such as a limited thickness, poorer through-thickness mechanical properties compared to rigid preforms, and are less performant in shear environments.

In this work, we used tomography reconstructions to analyze and compare the material properties and oxidation response of rigid and flexible substrates that are used to engineer lightweight carbon/phenolic ablators. For this class of materials with a small phenolic content, the substrate drives the thermal properties of the composite. Hence, a better knowledge of the preform architecture and properties can lead to improvements and optimization of the thermal protection material. Moreover, past investigations have shown that for PICA-like ablators the more reactive charred matrix (due to the high surface area) decomposes faster than the preform under the effect of oxygen reactions, and the fiber of the substrate are left exposed to the incoming flow [6,7]. Therefore, quantifying the oxidation of preform materials is important for predicting the overall heatshield performance.

The paper is structured as follows. Section 2 provides a review of the relevant literature on the use of micro-CT for heat and mass transport computations in material structures. In Section 3, we outline the use of synchrotron X-ray micro-tomography (micro-CT) to resolve the architecture of both felts and rigid preform at micron scale. Compared to a standard characterization obtained by scanning electron microscopy, micro-CT adds the possibility of resolving the material in three dimensions (3D) on voxels, and provides a digital representation of the material that can be used for numerical simulations. In Section 4, a commercial toolbox was used to perform computations of materials properties, based on micro-CT digital representations of the material. We focus on diffusivity and room temperature thermal conductivity, which are important properties for ablator response simulations. Micro-CT data also enable simulations of the mass transport within the micro-structure and computations of the material permeability. This is not considered here, however we refer to a companion work on the topic [8], where we showed that Klinkenberg permeability in slip regime can be computed from micro-CT, using the direct simulation Monte Carlo method, with excellent agreement against experimental measurements [9]. In Section 5, we used an in-house developed software, PuMA (Porous Materials Analysis) [10], to study the degradation of the material through oxidation. The paper conclusions (Section 6) provide an outlook on the prospective use of micro-CT for computing material properties, and the response of high temperature fibrous insulations to their environment.

2. Literature review

In this work, numerical simulations were performed using 3D images from X-ray micro-CT as computational domains. The past two decades have witnessed the emergence of micro-CT as a primary characterization tool in material science. This is due to its ability to non-destructively characterize a material in 3D, while attaining spatial resolutions ranging from sub-micron to centimeter scale [11–14]. Both synchrotron and laboratory sources have become established methods of producing X-rays for micro-CT imaging. As the technique advanced, so too did the development of computational methods that use the digital representation of a micro-structure offered by micro-CT in order to compute material properties and response.

The numerical determination of the effective properties of porous media and composites from the properties of the constituting phases (e.g. the effective thermal conductivity as a function of constituents' conductivities) has been an extensively explored field for nearly a century. The works of Torquato [15,16], Whitaker [17], Wang and Pan [18], and references therein are only a limited portion of the pertinent literature. While several computational and theoretical/analytical approaches have proven valuable in multiple applications, a clear advantage of micro-CT is that access to the real microstructure over a broad range of scales is provided.

Several authors have used tomography data to simulate heat transfer in porous media [19–33]. While focused on very different applications of material science, such as composites, porous ceramics, wood, packed beds, snow, etc., most of these studies aimed to characterize the material's effective conductivity. Only a limited number of them accounted for contributions of both conductive and radiative transfer in porous structures [21,23,24,29], and few of the published works focused on TPS applications, such as carbon/carbon [19] or silica/phenolic [30].

Micro-CT data have been used to compute permeability of porous structures [8,20,34–44] using Darcy's law under laminar, viscous, and continuum flow conditions. Some of these efforts considered modifications of Darcy's law to account for turbulent, shear and rarefied flow effects (Forchheimer, Brinkman, and Klinkenberg).

Numerous computations of diffusional transport and tortuosity [20,45–49] are also found in the literature, focusing on the determination of effective diffusion coefficients under rarefied (high Knudsen number[1]), continuum (low Knudsen number) or transitional regime (Bosanquet approximation). Among those studies, the works that focused on fibrous TPS materials are those by Vignoles et al. [20,46] on carbon/carbon composites, by White et al. [44] on carbon/phenolic ablative materials and by our group on fibrous insulators [8].

3. Micro-tomography imaging and materials

Micro-CT measurements were performed at the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory. Synchrotron X-rays produced by the ALS beamline 8.3.2 provide images at sub-micron resolution with low noise. This capability is ideal for resolving the fibrous structure of highly porous substrates that have porous characteristic lengths of the order of tens of microns.

The tomography setup and its capabilities were described in details by MacDowell et al. [50]. In this study, tomography images were collected using a 2560 × 2160 pixels pco.edge 5.5 sCMOS camera (PCO, Kelheim, Germany) and an Optique Peter microscope (Optique Peter, Lentilly, France), equipped with a 10× Olympus Plan Apo objective (Mitutoyo, Kawasaki, Japan) installed behind a 20 μm LuAG scintillator. The optics provided a pixel size of 0.645 μm. All of the measurements were performed using a monochromatic X-ray beam at 18 keV, collecting a total of 1024 radiographs in each scan. Micro-CT projections were reconstructed into 3D images using the Octopus software (inCT, Aalst, Belgium) [51], and an ALS in-house interface implemented in the Fiji software [52]. Fiji was also used to filter tomography artifacts and segment the images for visualization.

We imaged samples of rigid carbon preform, carbon felt and rayon felt. For the rigid material, we selected FiberForm™ from Fiber Materials Inc. (Biddeford, ME, USA), the substrate used in PICA. FiberForm is made from a slurry of chopped rayon-based carbon fibers mixed with phenol-formaldehyde resin and water. The slurry is vacuum casted, compressed and cured at high temperature into desired shapes. Of the conformal and flexible materials being developed at NASA Ames Research Center (ARC) [2], two different felts were considered. We analyzed a low-density rayon-

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[1] It is recalled that the Knudsen number is the ratio of the flow mean free path λ to the characteristic length of the medium L, which for porous materials it often assumed to be the mean pore diameter d_p.