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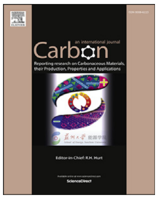

Modeling the oxidation of low-density carbon fiber material based on micro-tomography

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ABSTRACT

Oxidation is one of the main decomposition mechanisms of fibrous carbon/phenolic ablators employed in thermal protection systems for planetary entry capsules. The oxidation process is driven by two competing mechanisms: diffusion of reactants within the porous medium, and reaction rates at the surface of the fibers. These mechanisms are characterized by the Thiele number. Given that the Thiele number varies during an atmospheric entry, we aim to understand the effects of the diffusion/reaction processes on the decomposition of a porous carbon material in various regimes. We use a particle method for simulations of the oxidation process at microscale. The movement of oxygen reactants is simulated using a Brownian motion technique, and heterogeneous first-order reactions at the surface are modeled with a sticking probability law. To enable simulations of the fiber decomposition on actual materials, we use digitized computational grids obtained using X-ray micro-tomographic imaging. We present results for the oxidation of the substrate of the material used on the Mars Science Laboratory capsule that landed the Curiosity rover. We find that the depth of the reaction zone for this material is critically dependent on the Thiele number.

1. Introduction

To reduce the cost of access to space and to address the challenges that will be faced by future exploration missions, NASA is optimizing thermal protection system (TPS) materials for atmospheric entry vehicles. Low-density carbon/phenolic ablators, built upon a rigid carbon fiber preform impregnated with phenolic resin, have proven to be a successful class of TPS materials for exploration missions. The flagship architecture within this class is the Phenolic-Impregnated Carbon Ablator (PICA). This material was successfully used on the Mars Science Laboratory (MSL) and Stardust missions.

Carbon/phenolic ablators are designed to blow pyrolysis gases from the decomposing phenolic into the boundary layer formed at the surface of the material. The pyrolysis process mitigates part of the heat flux coming from the freestream plasma. Within the material, the fully charred (pyrolyzed) phenolic phase leaves a carbonized matrix. The carbonaceous remains, composed of carbon preform and charred phenolic, interact with the reactive species in the boundary layer through heterogeneous reactions. The main material recession processes in air are heterogeneous oxidation ($C_{(s)} + O \rightarrow CO$, $C_{(s)} + O_2 \rightarrow CO_2$), phase changes (e.g. sublimation $C_{(s)} \rightarrow C$), and mechanical erosion by friction and shear stress (referred to as spallation). In this work we focus on heterogeneous oxidation processes. These are chief contributors to the exothermicity of the ablation zone, especially at the surface of the TPS, where the highly reactive matrix leaves the carbon preform exposed to incoming oxidants.

For highly porous ablators, where the reacting gases can percolate in-depth, it is important to consider the oxidation phenomenon at the scale of the fibers in order to understand the competing effects of diffusional mass transport and gas/surface reactions. A suitable approach for the analysis of this problem was proposed by Lachaud and Vignoles [1]. The same method was applied to study oxidation of carbon/phenolic ablators using artificially generated materials [2]. These studies used simplified digital models that describe the statistical, three-dimensional (3D) morphology of composite materials. Lachaud and Vignoles' method

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is based on a representation of the average gas diffusion by random walks, a sticking probability law for modeling heterogeneous reactions and a simplified marching cube algorithm for tracking the moving surface [1].

Using synthetic material models to mimic the microstructure of real composites has some limitations. Features of real materials, such as complex fiber morphologies, fiber clusters, actual pore-size distribution and other irregular characteristics, are very difficult to describe analytically and are not captured by ideal geometries. Synchrotron X-ray micro-tomography (micro-CT), used in the past to image C/C composites [3], was recently applied to image substrates of carbon/phenolic ablators [4,5]. The technique provides a high-fidelity digital representation of the actual material microstructure where the geometry of the fibers is captured as a 3D matrix of grayscale values. Modern micro-CT instruments can reach voxel resolutions below a micrometer, thus are able to resolve the fibrous structures of carbon preform material in detail. The grayscale value within a voxel is proportional to the absorption of X-rays by the material, hence to the material density.

In this paper we aim to develop an understanding of the recession and in-depth evolution of the porosity of carbon fiber composites. We simulate the oxidation of FiberForm™ (Fiber Materials Inc.), a porous carbon fiber material made for commercial uses as furnace liners and for other applications. NASA has adopted FiberForm as the substrate for PICA. We refined the particle-based method described in Ref. [1], combined with high resolution micro-CT scans of the material, to study the oxidation of this substrate. The tomographic images of FiberForm were collected at the hard X-ray micro-CT beamline of the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory (LBNL). They consist of large datasets of the order of several billion voxels. In section 2, we describe the method of using micro-CT to develop a representation of the material on a three-dimensional Cartesian grid. These large datasets require dedicated tools for use in numerical simulations. To achieve this goal we have developed a computational framework, called *PuMA* for *Porous Materials Analysis*, described in section 3. The framework enables microscale simulations and calculations of certain material properties based on large micro-CT files.

In section 3.1, we describe the particle-based oxidation model. In section 3.2, we use analytical solutions for the oxidation of single fibers to verify the analysis tool. In section 4, we study the oxidation of the fibrous material at various Thiele numbers (or regimes). In section 5, we conclude that the competing mechanisms of diffusion through the porous material and gas/surface reactions control the depth of the reaction volume, and propose future directions for research.

2. Micro-tomography and visualization

We used 14 keV X-rays generated by the ALS synchrotron to collect micro-CT projections of FiberForm. We adopted the ALS in-house procedure for tomographic reconstruction [6]. The digitized geometry of the material consists of grayscale voxels that are effectively a 3D X-ray image of the material that needs to be further interpreted. Fig. 1 shows a surface rendering of the fibers in a 0.14 mm^3 volume. The visualization was obtained by using OpenGL to render an approximation of the isosurface of the fiber geometry based on the marching cubes algorithm [7,8].

The marching cubes algorithm is a level set method that effectively computes an isolevel surface of the grayscale levels. To select the cutoff level (or grayscale threshold, Γ^*) we start by computing a histogram of the grayscale distribution. Fig. 2a shows the histogram for the micro-CT image of FiberForm presented in Fig. 1. For this material, we found that the optimal grayscale cutoff is approximately the point of inflection on the histogram. This choice was confirmed by a quantitative comparison of the computed porosity from the image with the value specified by the material manufacturer, and a qualitative comparison of the tomography visualization with scanning electron microscopy images of virgin FiberForm. Any vertex with a grayscale value above this threshold is considered to be within the material, and any vertex below is considered void. An example of segmentation for a *x-y* slice of the tomography is shown in Fig. 2b (original) and Fig. 2c (thresholded).

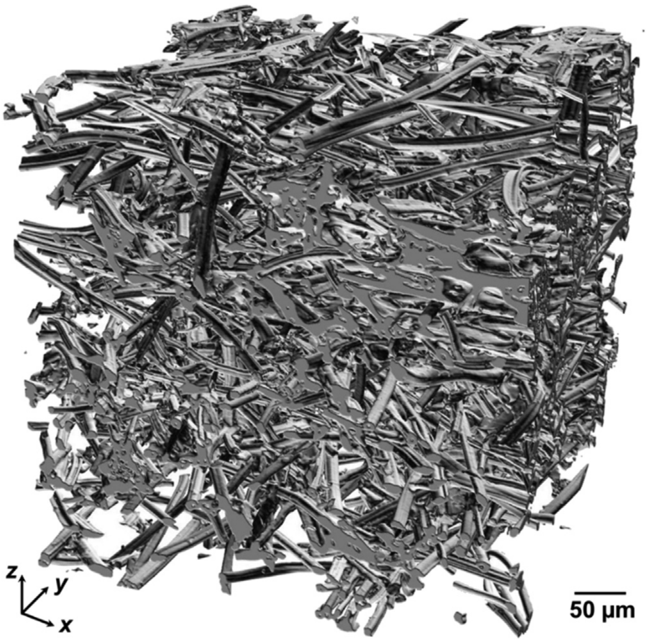

Fig. 1. Volume rendering of FiberForm micro-CT. The image displays a 0.14 mm^3 cube with approximately 0.325 μm voxel edge size (20× magnification). The rendering highlights the orthotropic structure of FiberForm, with fibers preferentially aligned within the *x-y* plane. Fiber clusters and other irregular features are appropriately resolved.

The marching cubes algorithm compares each voxel in the domain (represented by its 8 vertices) to a table of 256 possible polygon configurations, in order to determine which of the edge cases applies [7,8]. The edge intersections of each triangle are then linearly interpolated from the vertex grayscale values. The resulting collection of triangles can be used for visualization as well as for computation of the model specific surface area.

To verify our implementation of the marching cubes algorithm in the *PuMA* framework, we generated grayscale images of analytical objects, and compared the computed surface area of their isosurface (calculated as a sum of the individual triangle areas) with the analytical surface area of these 3D shapes (e.g. cylinders and spheres).

3. Microscale oxidation model and verification

3.1. Random walk oxidation

3.1.1. Digitization the micro-CT image of the material

In the previous section we identified the fiber surface by defining a grayscale cutoff level. To digitize the geometry of the fibers, the 3D tomography array containing the grayscale values is processed as follows: 1) if a fiber surface passes through a voxel, the grayscale values of that voxel remain unchanged; 2) if the voxel is either completely outside or completely enclosed in the material, the voxel vertices are set to 0 or 255, respectively. A schematic example of this processing is shown in Fig. 3, for a two-dimensional