

4-D MICRO-CT ANALYSIS OF SPACECRAFT HEAT SHIELD MATERIALS UNDER ATMOSPHERIC ENTRY CONDITIONS

Harold S. Barnard ^{*1}, Christian M. Schlepütz ^{†2}, Dilworth Y. Parkinson¹, Edward S. Barnard³, Alastair A. MacDowell¹, Nagi N. Mansour⁴, and Francesco Panerai ^{‡5}

¹Advanced Light Source, Lawrence Berkeley National Laboratory, USA

²Swiss Light Source, Paul Scherrer Institut, Villigen PSI, Switzerland

³Molecular Foundry, Lawrence Berkeley National Laboratory, USA

⁴Advanced Supercomputing Division, NASA Ames Research Center, USA

⁵Department of Aerospace Engineering, University of Illinois at Urbana-Champaign, USA

Keywords: high-speed X-ray micro tomography, 4D imaging, in-situ evolution of dynamic system, high temperature, heatshield, aerospace

Summary: We demonstrate an application of time-resolved X-ray tomography to study the oxidation of a porous carbon fiber material used in the heatshields of hypersonic spacecraft. An in-situ high-temperature setup was built to enable oxidation at controlled conditions with fast 3D microscopic imaging. It is shown that oxidation can be resolved with unprecedented details in both diffusion- and reaction-limited regimes.

1. INTRODUCTION

Spacecraft returning to Earth or entering the atmosphere's of other planets experience extreme heat loads due their high kinetic energy being dissipated through aerodynamic drag, often as high as 10 MW/m² for 10s of seconds, with peak temperatures exceeding 3000 °C. Spacecraft therefore require reliable and robust thermal protection systems (TPS) to shield the payload from these extreme aerothermal conditions. Lightweight carbon fiber ablators are preferred TPS materials because they are light weight and combine the high temperature performance of carbon with superior insulation capabilities.

A critical element in the design and sizing of carbon fiber ablative heat shields is predicting the oxidation behavior of the material. Efforts are underway to develop advanced computational models for carbon ablation that account for oxidation kinetics at different flight conditions. In past investigation we have shown that carbon fibers oxidation can be modeled upon microstructure data obtained from X-ray micro-tomography (μ CT). [4, 1]. In highly porous ($> 85\%$) materials the oxidation is controlled by competing reaction/diffusion processes. At high temperatures, oxidation is faster than diffusion which makes ablation a surface phenomenon. Conversely, at lower temperatures diffusion time scales become smaller, reactants diffuse in the porous medium prior to reacting with the fibers and the material decomposes in depth.

In this work, we apply fast X-ray micro-tomography to resolve the oxidation of FiberForm at microscopic scale. FiberForm is a rigid carbon fiber preform used as the backbone of NASA's low density carbon-phenolic ablators. An in-situ lamp-heated furnace was designed to house a capillary micro-reactor able to maintain the FiberForm material under controlled pressure and temperature conditions, while allowing for fast rotation of the reactor in the tomography stage.

2. EXPERIMENTAL METHOD

The experiments were performed at the TOMCAT Beamline at the Swiss Light Source (SLS) Synchrotron Facility using an environmental cell developed at the Advanced Light Source (ALS). This cell allows the sample ($\varnothing 2\text{ mm} \times 4\text{ mm}$) to be suspended in a $\varnothing 3\text{ mm}$ quartz capillary surrounded by a sealed 12 mm quartz tube to maintain a vacuum or controlled low to ambient pressure gas atmosphere [2]. The sample cell rotates on the

^{*}e-mail: hbar@lbl.gov

[†]e-mail: christian.schlepuetz@psi.ch

[‡]e-mail: fpanerai@illinois.edu

μ CT stage with the sample at the focal point of an array of six gold parabolic-reflector halogen heat lamps. This allows sample temperatures of $>1200^{\circ}\text{C}$ to be achieved (Fig. 1(a)). Temperature is monitored with an internal type-K thermocouple in direct contact with the sample while pressure is measured at the pumping outlet. The samples were exposed to high temperature atmospheric entry conditions while performing high-speed continuous μ CT analysis with the GigaFRoST X-ray camera system [3]. This allowed for imaging rates of 0.25 seconds/tomogram to generate 4D time resolved data sets of the material decomposition. Direct data readout by the GigaFRoST system, enabled continuous acquisition several hundred tomograms per experiment.

3. RESULTS

FiberForm samples were heated at 900, 1000, 1100 and 1200°C at pressures of 0.025, 0.25, 0.6, 1.0 atmospheres (air) while performing μ CT analysis. Rapid oxidation and decomposition of FiberForm was observed at unprecedented spatial and temporal resolution: $0.8\text{--}2.75\mu\text{m}/\text{pixel}$ with scan rates up to 4Hz, capturing ~ 100 μ CT time points per measurement. An example dataset is shown in Figure 1(b)). The data show a detailed depth evolution of the material oxidation as it penetrates the surface. Preliminary analysis shows that oxidation occurs primarily at the surface for the the highest temperatures: $1100\text{--}1200^{\circ}\text{C}$, which suggests that oxidation occurs at diffusion-limited regime at these conditions. At 900°C the oxidation spans the entire sample thickness, which is typical of a reaction-limited regime. It was observed that in-depth oxidation causes the material to loose its structural rigidity and to progressively collapse under its weight. It is anticipated that in an actual hypersonic environment, this bulk weakening of the material might lead to massive material spallation under the effect of high shear flows. The oxidation results observed from the presented data are consistent with the numerical predictions obtained with the NASA Porous Material Analysis (PuMA) software [4]. The data provide a unique set for validating numerical models and for extracting effective oxidation reaction rates to be used in design ablation calculations.

References

- [1] Panerai, Francesco, et al. "Micro-tomography based analysis of thermal conductivity, diffusivity and oxidation behavior of rigid and flexible fibrous insulators." *International Journal of Heat and Mass Transfer* 108 (2017): 801-811.
- [2] Barnard, Harold S., et al. "Synchrotron X-ray Micro Tomography at the ALS: In-Situ Sample Environments for Advanced Aerospace Materials." *Microscopy and Microanalysis* 24.S2 (2018): 444-445.
- [3] Mokso, Rajmund, et al. "GigaFRoST: the gigabit fast readout system for tomography." *Journal of synchrotron radiation* 24.6 (2017): 1250-1259.
- [4] Ferguson, Joseph C., et al. "Modeling the oxidation of low-density carbon fiber material based on micro-tomography." *Carbon* 96 (2016): 57-65.

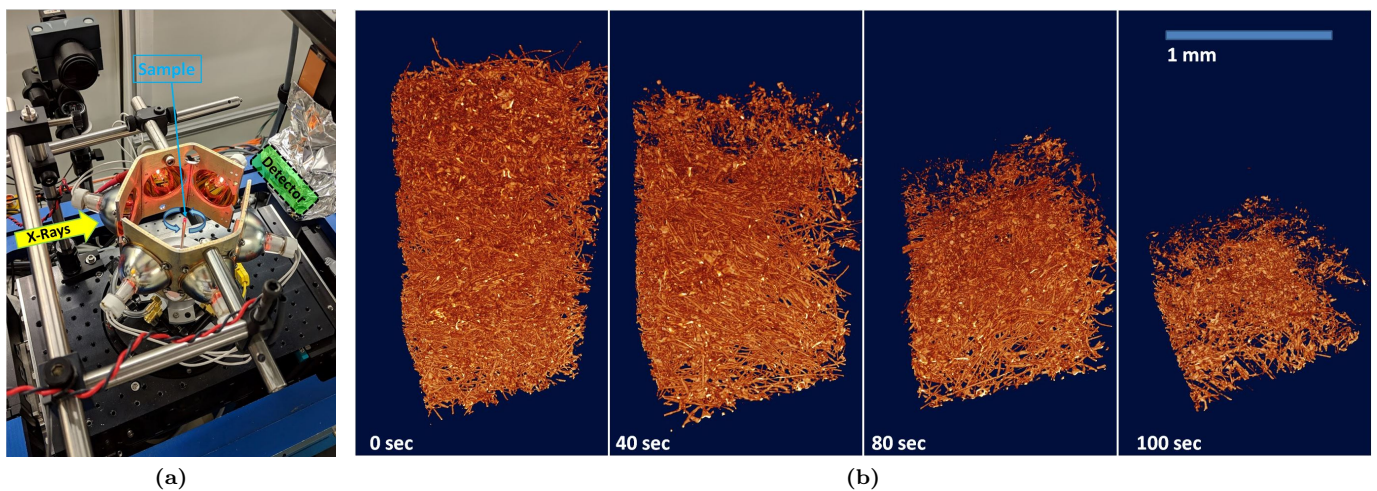


Figure 1: (a) In-situ environmental cell installed on tomography stage at SLS. (b) Four out of 67 μ CT time points spanning FiberForm oxidation at 900°C .