Flow-tube reactor experiments on the high temperature oxidation of carbon weaves

Francesco Panerai, Jason D. White, Robert Robertson, Arnaud Borner, Joseph C. Ferguson, Nagi N. Mansour

1–4NASA Ames Research Center, Moffett Field, CA 94035 – 5SRI International, Menlo Park, CA 94025 – 6STC at NASA Ames Research Center, Moffett Field, CA 94035

Overview
Under entry conditions carbon weaves used in thermal protection systems (TPS) decompose via oxidation. Modeling this phenomenon is challenging due to the different regimes encountered along a flight trajectory. Approaches using equilibrium chemistry may lead to over-estimated mass loss and recession at certain conditions. Concurrently, there is a shortcoming of experimental data on carbon weaves to enable development of improved models. In this work, a flow-tube test facility was used to measure the oxidation of carbon weaves at temperatures up to 1500 K. The material tested was the 3D carbon weave used for the heat shield of the NASA Adaptive Deployable Entry and Placement Technology, ADEPT [1]. Oxidation was characterized by quantifying decomposition gases (CO and CO₂), by mass measurements, and by microscale surface analysis. The current set of measurements contributes to the development of finite rate chemistry models for carbon fabrics used in woven TPS materials.

Experiment
Experiments were performed at the flow tube reactor facility at SRI [2]. The facility consists of a furnace-heated, vacuum-pumped quartz tube, equipped with a series of mass flow meters, pressure transducers, and temperature controllers (Figure 1). Inert gases, used during heating and cooling, and oxidizers (O₂ and CO₂ in the present work) are flown at controlled rates, while measuring the environmental pressures.

Materials characterization
Scanning electron microscopy (SEM) images of the samples prior to and after oxidation revealed:

- smooth surface for the PAN-based fibers of the virgin material;
- oxidation by pitting of the fibers’ surface, similar to the oxidation of rayon-based fibers observed in previous investigations [3];
- increased pitting with temperature, leading to fibers thinning and loss of tow compactness;
- incremental fraying of the woven structure that indicates ease for the shear flow to remove material by mechanical erosion [4].

Conclusions & outlook
Progress has been made in quantifying the oxidation of 3D carbon weaves being developed for new generation NASA TPS. Experimental observations and microscale analyses showed a significant decomposition of the weaves by mechanical removal of oxidized fibers in weakened tows. Future efforts will be focused on expanding the study to the oxidation and nitridation of carbon weaves under the effect of O₂ and N₂atoms.

Results
Experiments were carried out at several temperatures in O₂ and CO₂ for approximately 10 minutes oxidation time. O₂ tests yielded increasing mass loss up to 60% for the highest temperature. Negligible loss was measured in CO₂.

Quantification of oxidation products confirmed CO₂ production at low temperatures and increasing CO production at increasing temperature.

Quantification of mass loss and oxidation products during O₂ tests. Mass spectrometry measurement of gaseous decomposition products during oxidation revealed predominant CO₂ production at low temperatures. At temperatures above 1100 K, CO prevailed instead. No O₂ consumption was observed at temperatures below 800 K.

References

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