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14. ABSTRACT
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Accurate modeling of these conditions requires thermophysical properties and deposition parameters for carbonaceous deposits. Previous studies have demonstrated multilayer deposition in fuel-film cooled combustors likely occurring due to a combination of heterogeneous condensation of polycyclic aromatic hydrocarbons and thermophoretic diffusion of combustion soot [1]. Figure 1 shows a typical sample and distinguishes dense from soot layers. Differences in chemical and physical structure cause the condensed (abbreviated to dense) and soot deposit layers to have different properties. Therefore, all relevant thermophysical properties will be required for both layers to develop accurate heat transfer models.

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Carbonaceous Deposit Density in a Fuel-Film Cooled Rocket Combustor

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I. Introduction

Hydrocarbon fuel-film cooled rocket combustors develop carbonaceous deposits that insulate regenerative cooling jackets from the chamber hot gases. These deposits are formed in conditions atypical for combustion soot literature: at pressures over 5 MPa, in forced convection flows with $Ma > 0.2$ gaseous core flows, and on highly cooled walls with heat fluxes over 10 MW/m^2.

Accurate modeling of these conditions requires thermophysical properties and deposition parameters for carbonaceous deposits. Previous studies have demonstrated multilayer deposition in fuel-film cooled combustors likely occurring due to a combination of heterogeneous condensation of polycyclic aromatic hydrocarbons and thermophoretic diffusion of combustion soot [1]. Figure 1 shows a typical sample and distinguishes dense from soot layers. Differences in chemical and physical structure cause the condensed (abbreviated to dense) and soot deposit layers to have different properties. Therefore, all relevant thermophysical properties will be required for both layers to develop accurate heat transfer models.

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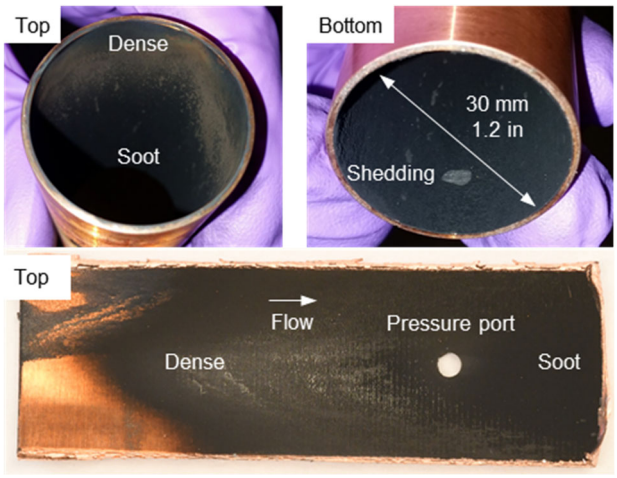

Fig 1 Post-test images of the inlet (top left) and outlet (top right) of a sample tube chamber liner. High resolution image of sectioned sample (bottom).

Graphite has a theoretical single-crystal density of 2266 kg/m^3, however typical engineering graphite has a density of around 1800 kg/m^3 due to its small crystallite size [2]. Soot agglomerate effective density from diesel engine exhaust was measured by Park et al. [3], with values ranging from 300 to 1600 kg/m^3. Larger agglomerate mobility diameters resulted in lower effective densities. Note that effective density is not the same as apparent or envelope density.

Density can be quantified in several different manners for powders and other materials with voids. Absolute density excludes the volume of both closed and open pores, and can be thought of as a measure of the density of only the solid portion of the powder. Envelope density includes the pore volume, and is therefore always lower in value compared to absolute density.

We measured densities of dense and soot layers formed in a fuel-film cooled H_2O_2-kerosene rocket combustor that operated at 4.8 MPa [4]. Apparent densities were measured with a pycnometer and envelope densities were measured with a scanning electron microscope (SEM) and optical profilometer (for layer depth and volume) as well as a high accuracy scale (for mass).

II. Combustor

The experimental hardware consisted of a kerosene-H_2O_2 bipropellant fuel-film cooled rocket combustor that operated at pressures up to 4.8 MPa and run times up to 15 s. The combustor was designed to have an axisymmetric heat sink sample chamber and removable chamber liners.

Figure 2 shows the fuel-film cooled combustor used for the tests outlined in this experimental effort. Flow through the combustor started with 90 wt.% hydrogen peroxide injected through a 25 mm inner diameter catalyst bed packed with silver-plated wire mesh screens to decompose the H_2O_2 to approximately 1030 K hot oxygen and steam. Fuel was radially injected through four 0.64 mm holes into the hot oxygen and steam flow to promote mixing at a stoichiometric mixture ratio of 7.5. A 0.38 mm thick yttria-stabilized zirconia thermal barrier coating protected the flame holder section of the combustor from the high enthalpy combustion gases, which had temperatures up to 2800 K when operated at stoichiometric conditions. Downstream of the step, a fuel-film injector introduced a low velocity annular kerosene flow through a 0.25 mm axisymmetric gap to cool the inner wall of an annular sample chamber liner. Sample chamber liners were thin, precision-machined tubes with inner and outer diameters of 29.8 mm and 32.0 mm respectively. The sample length of 74 mm was long enough that the fuel-film decomposition occurred on the sample. A graphite converging-diverging nozzle with a 1.14 cm throat diameter choked the flow to achieve chamber pressures up to 4.8 MPa.

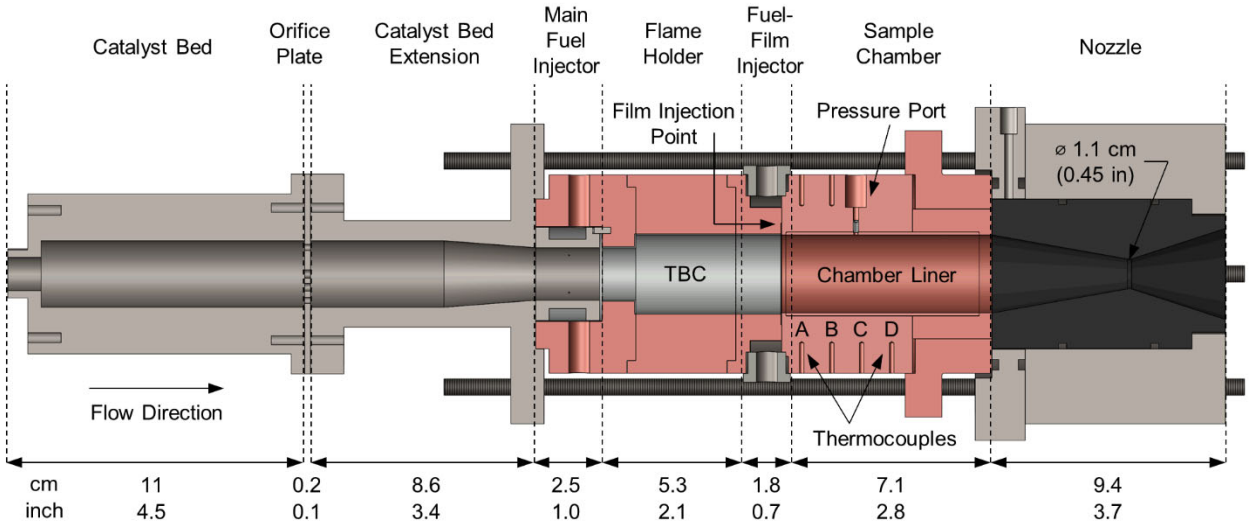

Fig. 2 CAD model of the fuel-film cooled kerosene-oxygen combustor used in this study.

III. Methods

A Micromeritics AccuPyc II 1340 gas pycnometer was used with helium to determine the apparent density of soot and dense carbon deposit powder samples. The pycnometer 3.5 cm^3 sample cup was calibrated with a metal sphere standard, as per the operator manual [5]. Sample collection involved lightly scraping soot deposits from chamber samples, requiring over 15 cm^2 of area to obtain accurate measurements. The dense deposits could not be removed in sufficient volume for gas pycnometry analysis.

In-situ envelope density, which accounted for soot volumes, was determined by measuring layer mass and layer volume. Layer mass was measured gravimetrically by progressively cleaning the soot and dense layers from each sample. Light cleaning with isopropyl alcohol removed the soot layer while leaving the dense layer mostly intact. However, the distinction between dense and soot layer can often be ambiguous, even when imaging with the SEM. For instance, some dense layer SEM cross sections showed multiple dense layers with air gaps between them, which would manifest as a lower total density when integrated across the entire sample surface area (see Fig 3). The more tenacious dense layer was removed by ultrasonic cleaning in Ensolv®, a n-propyl bromide solvent, for 60 minutes. Layer depth was measured by integrating SEM and optical profilometry depth measurements as a function of axial distance from the fuel-film injector. Layer volume could then be calculated by measuring the sample surface area and assuming an axisymmetric deposition profile. The soot layer volume was calculated by subtracting the dense layer volume (measured with SEM [6]) from the total volume (measured with profilometer [7]). The largest uncertainties in this analysis were from sample sectioning, soot layer cleaning, and the axisymmetric deposition profile assumption.

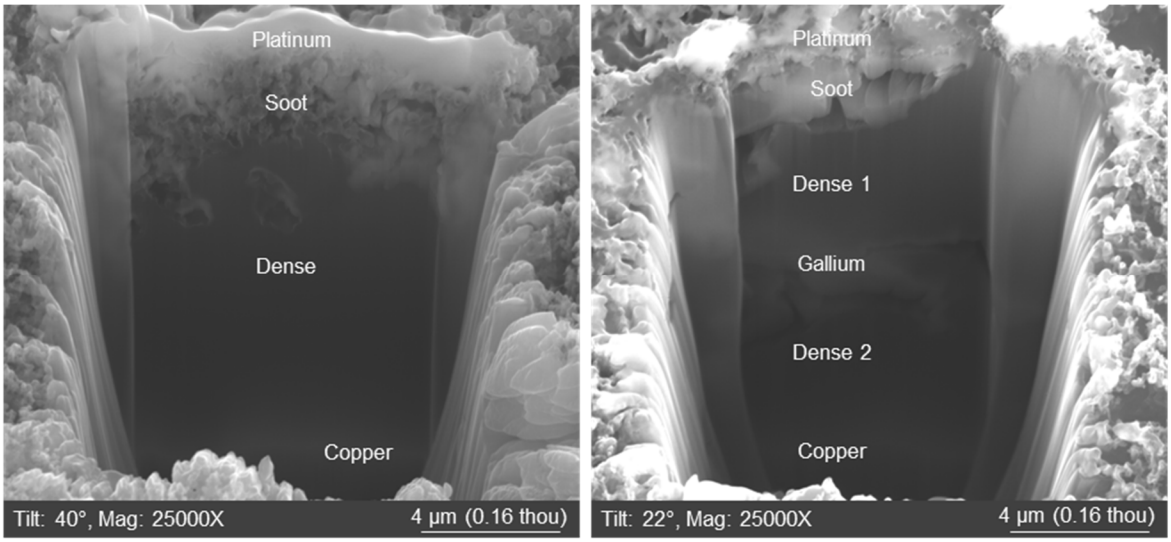

Fig 3 Representative well-defined (left) and ambiguous (right) soot-dense carbon layer transitions from two different axial positions of the same sample.

IV. Results

Apparent density measurements from tests 40, 58, and 63, which occurred at a variety of conditions (see Table A1), resulted in similar values to those expected for engineering graphite, as shown in Table 1. Apparent and envelope densities of the soot and dense layers were approximately constant across tests 57, 58, 60, and 63. Test 60 apparent density is not comparable with tests 57, 60, and 63 because the 4-6-4 test structure, with its four second monopropellant firing occurring after bipropellant shutdown, resulted in significant oxidation and removal of the soot layer. Similar data precision was noted for both the soot and dense layer envelope densities after neglecting T60. Dense layer envelope densities approached the apparent density of the soot layer, where the deviation occurred due to the dense-soot transition region and intra-dense-layer air gaps. The soot layer was almost an order of magnitude less dense than the dense layer, and is therefore expected to be the primary contributor to thermal insulation.

Table 1. Apparent and envelope densities of fuel-film cooled combustor carbonaceous deposits. Test 60 used a 4-6-4 test autosequence (see Appendix A1) and did not match conditions of the other tests.

Test Number	Soot Layer Apparent Density (kg/m^3)	Dense Layer Envelope Density (kg/m^3)	Soot Layer Envelope Density (kg/m^3)
40	1929	-	-
57	-	1336	202
58	1935	1639	244
60	-	1835	172
63	1874	1419	203

V. Conclusions

Apparent and envelope densities for multilayer carbonaceous deposits were measured using a gas pycnometer, scanning electron microscope, optical profilometer, and gravimetric analysis. The deposits were formed on annular metal sample tubes in a 4.8 MPa chamber pressure fuel film cooled rocket combustor. Apparent densities of 1900 kg/m3 for the soot upper layer matched those previously published in the combustion literature and approached values for engineering graphite. Soot envelope densities around 200 kg/m3 were significantly lower due to their highly porous structure. Dense layer envelope densities from 1336 to 1835 kg/m3 were close to the soot apparent density, with some variation due to ambiguous dense to soot layer transitions that the density measurement method could not distinguish. These data can be used for heat transfer modeling of hydrocarbon rocket combustors, where carbon deposition often insulates chamber walls.

Appendix

Table A1 Relevant portions of the fuel-film combustor test matrix. The full test matrix can be found in our previous publication [4].

Test Num	Test Date	Fuel	Biprop Time (s)	Biprop Pressure (MPa)	Core Mixture Ratio	Total Mixture Ratio	Sample Material	Sample Surface Roughness	Fuel-Film Mass Flow Rate (g/s)
40	06/18/2018	RP–2	15	4.83	7.44	2.89	C10100	Fine	52.5
57	03/13/2019	RP–2	6	4.62	7.48	3.52	S30400	50–50[b]	37.8
58	03/13/2019	RP–2	6	4.63	7.47	3.52	N06600	50–50[b]	37.8
60	04/17/2019	RP–2	6[a]	4.56	7.35	3.46	N06600	50–50[b]	37.8
63	04/29/2019	RP–2	10	4.58	7.36	3.47	N06600	50–50[b]	37.8

[a] A test that fired monopropellant mode both before and after bipropellant mode.
[b] Surface roughness of 50-50 indicates a sample tube chamber liner that had 50% of its interior artificially roughened.

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