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Ablation behavior of C/C-SiC-ZrB_2 composites in simulated solid rocket motor plumes

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ABSTRACT

In order to improve the ablation resistance of carbon/carbon (C/C) composites, SiC-ZrB_2 was infiltrated into them by reactive melt infiltration (RMI). Their ablation behavior with a leading edge shape was investigated in the combustion environment of a simulated solid rocket motor (SRM). The results show that after introducing SiC-ZrB_2 ceramic phases into C/C composites, the linear and volume ablation rate of modified leading edge decreased by 26% and 45%, respectively. The high erosion rate of the composites leading edges was mainly attributed to the synergistic effect of thermochemical ablation and mechanical erosion in a high-velocity, high particle concentration two-phase flow of SRM plume environment.

1. Introduction

Carbon fiber reinforced carbon matrix (C/C) composites are considered as potential high-temperature structure materials for engineering and aerospace applications due to their excellent and unique properties such as lower density, high specific strength-to-weight ratio, good chemical stability and resistance to thermal shock [1–3]. Unfortunately, these composites have poor oxidation and ablation resistance properties, because carbon will be oxidized over 500 °C, which restricts their application in aerospace fields. In order to expand the application area of C/C composites, preparing protective ultra-high temperature ceramics (UHTCs) coatings or modifying C/C composites by UHTC is a promising and effective approach. The service life, security and structural integrity of the modified composites have been tested in different high temperature environment including plasma wind tunnel, arc-jet, laser, oxyacetylene flame and methane wind tunnel [4–8]. The results show that UHTC-C/C composites have an excellent mechanical property, good oxidation and ablation resistance at high temperature.

It has been certified that UHTC-C/C composites containing a single ceramic phase could not effectively resist the erosion of high temperature flame, and multilayered or multiphase UHTC-C/C composites had better oxidation and ablation resistance [9–11]. Among them, C/C-SiC-ZrB_2 composites have attracted extensive attention due to their superior properties for ultra-high temperature application [12–14], such as thermal protection systems, exhaust-section flaps/seals and afterburners of rocket engines. In order to ensure the security and stability of spacecraft, several methods have been employed to test their ablation and oxidation resistance in high temperature environment [15–20]. The results show that SiC and ZrB_2 ceramic phases are oxidized at high temperatures, forming a complex oxide scale consisting of zirconia skeleton, which act as protection barriers against oxidation and ablation.

For the erosion of the composites, previous studies focused primarily on thermal chemical ablation (i.e., thermal decomposition and thermal erosion) due to the flame with high temperature and speed. Actually, the composites not only were eroded by high temperature flame but also attacked by fine particles from the solid propellant. The particles with elevated temperature and high relative speed, collectively referred to as CMAS (calcia-magnesia-alumino-silicate), impact on the surface of composites in high temperature environment causing them to fail prematurely. However, there has been little research reported the ablation behavior of

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$C/C-SiC-ZrB_2$ composites under such extreme conditions. It is necessary and meaningful to clarify the ablation mechanism of the sharp $C/C-SiC-ZrB_2$ composites exposed to ground simulated high speed, high temperature and high particle concentrated two-phase flow conditions, extending the application of $C/C-SiC-ZrB_2$ composites in aerospace field.

In this work, wedge-shaped $C/C-SiC-ZrB_2$ composites were prepared by reactive melt infiltration process. In order to simulate the severe operating conditions–higher temperature, faster speeds, hostile environments, a special lab-scale solid rocket motor (SRM) was used to evaluate the ablation resistance of these composites. The erosion morphology, microstructure and mechanisms of the $C/C-SiC-ZrB_2$ composites were discussed.

2. Experimental

2.1. Material preparation

2D carbon fiber felts with a density of 0.45 g/cm^3 were fabricated by alternatively stacked non-woven layers and carbon fiber webs by a needle-punching technique, which were employed as reinforcement for C/C composites (Fig. 1a). Methane gas was used as matrix precursor. Porous C/C composites with the density of 1.20 g/cm^3 were obtained through a low pressure chemical vapor infiltration process in the range of 900–1100 °C (Fig. 1b). The C/C preforms were machined into lab-scale leading edges, as shown in Fig. 2. The axial orientation of leading edge was parallel to *Z* direction of the preform, while the radial orientation was parallel to the *X-Y* plane of the preform. Then the porous C/C composites were embedded in the pack mixture, 18–25 wt% (300 mesh) Si, 20–25 wt% (200 mesh) B_4C, 45–55 wt% (200 mesh) $ZrSi_2$, and 5–10 wt% (300 mesh) C in a graphite crucible, and put into an electric furnace. The furnace was heated up to 1900–2100 °C with the heating rate of 15 °C/min and kept at this temperature for 2 h, then was cooled down to room temperature in argon atmosphere. Finally, the $C/C-SiC-ZrB_2$ composites were obtained (Fig. 1c), in which the SiC and ZrB_2 were introduced into the porous C/C composites by reactive melt infiltration process. For comparison, C/C composites were also prepared by low pressure chemical vapor infiltration process simultaneously. The apparent density and open porosity of the composites were 2.30 g/cm^3 and 7.5% for $C/C-SiC-ZrB_2$ composites and 1.75 g/cm^3 and 9.2% for C/C composites, which were measured by Archimedes method.

2.2. Erosion test

The erosion test was accomplished by a hot-firing test using a downsized SRM. The hot firing test system mainly consisted of nozzle, composite samples, combustion chamber, solid propellant and steel case. The sample was exposed to the SRM plume, as shown in Fig. 3a. Centerline of the sample was aligned with the axis

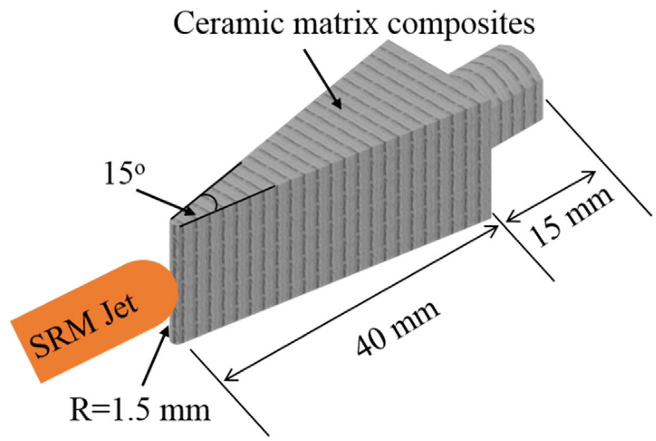

Fig. 2. Schematic of the leading edge sample.

of the nozzle. The distance between the leading edge and the exit plane of the nozzle was 20 mm. The SRM was loaded with the composite propellant, which was composed of 69.5 wt % ammonium perchlorate (AP), 12.1 wt % hydroxyl-terminated polybutadiene (HTPB), and 17.5 wt % aluminum powder and some other additives. The theoretical flame temperature of the propellant was about 3200 °C. The nominal chamber pressure was about 7.0 MPa. Finally the coupon was exposed to the plume for 5–7 s test durations. The screenshot for the video recording of sample in SRM plume is shown in Fig. 3b.

In order to simplify the pressure and mass calculations in the combustion chamber, several assumptions were adopted as followings:

a) Burning area of propellant remained constant;
b) The pressure and temperature of combustion gas distribution in combustion chamber was uniform;
c) The dimension of nozzle throat and leading edge was constant;
d) Combustion gases (not including Al_2O_3 particles) could be regarded as ideal gases;
e) The concentration of Al_2O_3 particles in combustion chamber was uniform, and its initial velocity was zero.

The equilibrium pressure of combustion chamber, p_c, was given by Ref. [21]:

$$p_c = \sqrt[1-n]{\frac{\rho_b c^* a A_b}{A_t}} \quad (1)$$

where ρ_b was the density of propellant, c^* was the characteristic velocity of combustion gases, A_b was burning area of propellant, A_t

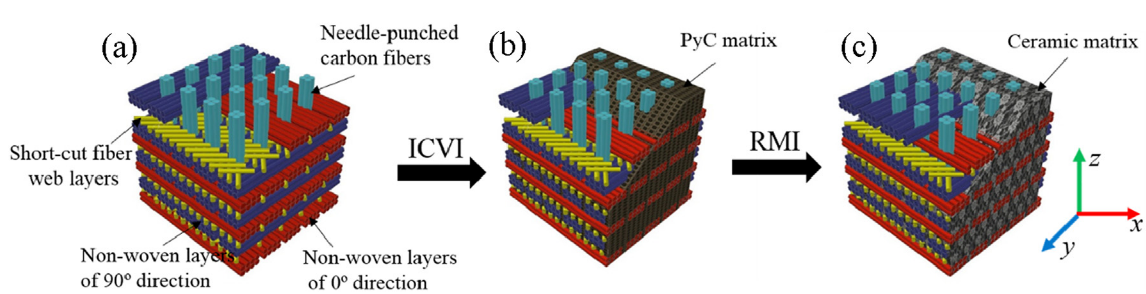

Fig. 1. Schematic of the preparation process of the composites: (a) 2D carbon fiber felt; (b) porous C/C composites; (c) ceramic matrix composites.