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Carbon fiber reinforced silicon carbide composite-based sharp leading edges in high enthalpy plasma flows

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ABSTRACT

Carbon fiber-reinforced silicon carbide (C/SiC) and zirconium diboride-zirconium carbide (ZrB_2-ZrC) modified C/SiC (C/SiC-ZrB_2-ZrC) sharp leading edges (SLEs) were prepared in this study. Their ablation behaviors were tested in plasma wind tunnels. The results indicated that the ablation of C/SiC SLEs was controlled by the atomic oxidation of SiC, leading to the elimination of SiC. In contrast, C/SiC-ZrB_2-ZrC SLEs showed little reduction in plasma flows. During ablation, a ZrO_2 skeleton was formed with silica filling inside by the atomic oxidation of SiC. The ZrO_2 skeleton suppressed the consumption of SiC, resulting in the improvement of the ablation resistance of C/SiC-ZrB_2-ZrC SLEs.

1. Introduction

In order to develop hypersonic vehicle with faster speed and higher flying altitude, the aerodynamic characteristics should be considered first [1–7]. The design of hypersonic vehicles requires sharp leading edges (SLEs) with millimeter scale radii of curvature (R) [2–9]. The sharp configuration helps the vehicle to improve its performance and maneuverability by reducing the aerodynamic drag and could provide a significant increase in the aerodynamic efficiency of the vehicle during flight [1,2,5]. However, the current challenge related to the SLE technology is the convective heating of the surface, a decisive factor for the surface temperature, which increases dramatically with the decrease in the value of R of the SLE [2,10,11]. The heat flux at the stagnation point exhibits an inverse dependence on the square root of R when R is less than 5 mm [2,12]. The materials for SLEs structure have to withstand more complex and severe environment than ordinary thermal protection component ablation [5,9,13–15]. Therefore, screening of materials for such applications has drawn more attention than ever before.

Carbon fiber-reinforced silicon carbide (C/SiC) composites composed of SiC matrix, pyrolytic carbon interface, and carbon fiber are considered as one of the promising candidate materials for SLEs structure, due to their low density, good high-temperature mechanical properties, and oxidation resistance [16–26]. With the decrease in the value of R of SLE, the heat flux at the stagnation point could reach an extremely high value. In this case, the C/SiC composite may not withstand the ablation in severe environments [19,21]. In general, the ultrahigh temperature ceramic (UHTC) components, such as zirconium diboride (ZrB_2) [27], zirconium carbide (ZrC) [28], hafnium carbide (HfC) [29], hafnium diboride (HfB_2) [30], tantalum carbide (TaC) [31], or their combination, would be introduced into the matrix or coatings to improve the temperature tolerance, which was confirmed by the ablation test under oxyacetylene torch [19,27,32] or laser beams [33,34]. However, the ablation of SLEs experiences extremely complex chemical processes in the real serving environments, such as re-entry from low earth orbit [2,13,29]. These processes also include the decomposition and recombination of gaseous species and the resultant mass recession other than only the high temperature [2,35,36]. Among the ground tests for ablation, the arc-jet and plasma wind tunnel are the best approaches to simulate the serving condition in the aerospace [5,10,35–38]. Compared to the arc-jet method, plasma wind tunnel technique could provide high enthalpy and purer plasma flows with a relatively low experimental cost [5,35,37]. Therefore, plasma wind tunnel is extensively employed for the materials' screening at the ground level.

Understanding the ablation mechanism is crucial for selection of the materials for SLEs under different conditions. Unfortunately, the

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C/SiC and modified C/SiC composites in the close serving environments have rarely been investigated, even though many ablation studies have been performed by using oxyacetylene torch [21,39,40], laser beams [33,34], or combustion chamber [41]. In this study, the ablation behaviors of C/SiC and C/SiC-ZrB_2-ZrC SLEs in the plasma wind tunnel were systematically investigated. The ablation process in plasma flows was revealed based on the analysis of the formed oxide on the composites after tests. The results of this study may provide a new perspective to the better understanding of the function of UHTC components in the modified C/SiC composites for applications in SLE.

2. Experimental procedures

A T-300TM three-dimensional needle-punched felt was fabricated by needle-punching technique with alternatively stacked weftless piles and short-cut-fiber webs (fiber volume ~35%). Pyrolytic carbon layer with a thickness of ~100 nm was deposited to obtain a weak interphase. The chemical vapor infiltration (CVI) method was then used to densify the felt using methyltrichlorosilane (MTS, CH_3SiCl_3) as precursor [42]. The density of the porous C/SiC preform was controlled to be approximately 1.4–1.5 g cm^{-3} with an open porosity of ~25%. For the plasma wind tunnel test, the obtained C/SiC preforms were machined into a SLE with $R = 1.5$ mm. The length and width of SLEs preforms were 80 and 25 mm, respectively, with 12° oblique angle and $\Phi 10 \times 10$ mm clamped cylinder.

The obtained C/SiC SLE preforms were divided into following two categories: pure C/SiC SLEs and C/SiC-ZrB_2-ZrC SLEs. The pure C/SiC SLEs were prepared by further densification of the C/SiC preforms by CVI-SiC. The final densities of the C/SiC SLEs were controlled to be 2.1–2.2 g cm^{-3} with an open porosity below 9%. The C/SiC-ZrB_2-ZrC SLEs were fabricated by reactive melt infiltration method as follows: The obtained C/SiC preforms were impregnated with the B_4C-phenol solution that was prepared by dissolving phenolic resin (PF, 2313, 96.5% purity, Xian resin factory, Xian, China) and B_4C (diameter 0.5 μm, 99% purity, Lihua Gaoke Chemical Company, Beijing, China) in ethanol, followed by curing at 150 °C for 5 h, and pyrolysis at 1500 °C for 2 h. The impregnation-pyrolysis process was repeated several times until the density of the preforms reached 1.6–1.7 g cm^{-3}. $ZrSi_2$ alloy powder (99% purity, Hai Chem. Co., Ltd., Beijing, China) was then melted at 1600–1700 °C, infiltrating into the preform to react with the pyrolytic carbon-B_4C residuals to form SiC-ZrB_2-ZrC matrix. The densities of the final C/SiC-ZrB_2-ZrC SLEs were controlled to be 2.3–2.4 g cm^{-3} with an open porosity also below 9%. The macro morphologies of the two types of samples are shown in Fig. 1. The C/SiC SLEs samples were covered with black SiC coating (Fig. 1a), while C/SiC-ZrB_2-ZrC SLEs were covered with silvery SiC-ZrB_2-ZrC coating (Fig. 1b).

Ablation experiments were performed in a 1 MW high-frequency plasmatron at Ultrahigh Speed Aerodynamics Research Institute, China (Aerodynamics Research and Development Center). The schematic illustration of the system and the detail description of the plasma wind tunnel have been reported in our previous studies [29,43]. It is a high enthalpy wind tunnel that generates plasma gas by electromagnetic induction, at sub-atmospheric pressure. The stagnation pressure P_d was measured by using a pitot tube. This was controlled within ±2% accuracy. The cold-wall heat flux q_{cw} was measured using a slug calorimeter with the same size as the test samples. A description of the measurement method could be found in the literatures [44–46]. During the tests, atmosphere was controlled to be standard air environment.

Density of the samples was measured by the Archimedes' method using distilled water. The mass of the samples before and after plasma exposure was measured using a balance with an accuracy of 0.01 g. During the ablation experiments, infrared and optical windows in the test chamber allowed for visual inspection and diagnostic analysis. The infrared pyrometer with ±20 °C accuracy was operated at 500–2500 °C. The microstructures of the samples before and after ablation were investigated by scanning electron microscopy (SEM, JEOL 6700F, Tokyo, Japan). The elemental analysis was conducted by energy dispersive spectroscopy (EDS) equipped with scanning electron microscope.

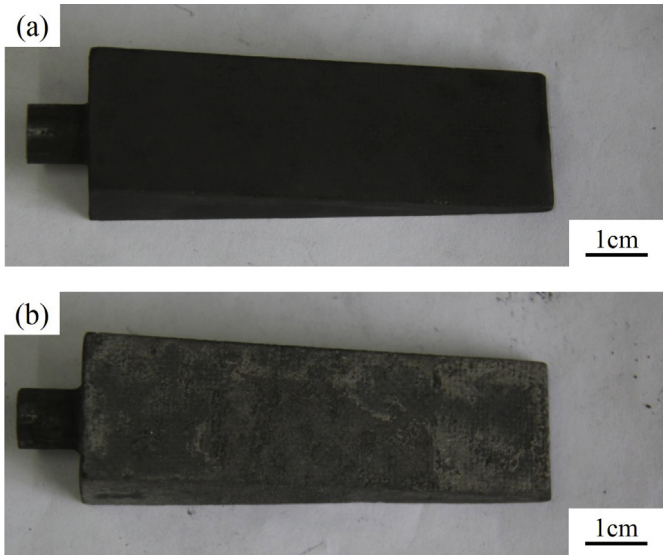

Fig. 1. The macro morphologies of the two types of samples.

3. Results and discussions

Three conditions were selected for the ablation of these SLE samples. The ablation lasted for 600 s in order to protect the test system. The corresponding samples for ablation were termed as CS-1–CS-3 for C/SiC SLEs and CZ-1–CZ-3 for C/SiC-ZrB_2-ZrC SLEs, respectively. The test conditions and the ablation results are listed in Table 1.

Fig. 2a exhibits the temperature profile as a function of ablation time of C/SiC SLEs. Clearly, the temperature reaches a steady state after ~20 s. Larger heat flux and stagnation pressure result in a higher steady temperature, ranging from 1373 to 1541 °C. The mass and linear recessions of the CS SLEs increase correspondingly with the increase in the steady temperature (Table 1). However, the change in the shape of CS SLE samples after ablation was limited based on their macro morphologies as shown in Fig. 2b. Only some color change and uneven topography appeared on the edge of CS SLE samples, which was caused by the ablation in high enthalpy flows.

The surface morphologies of the ablated CS SLEs samples at the stagnation point area are shown in Fig. 3. A protective homogeneous layer covers the CS-1 sample after ablation without carbon fiber exposure (Fig. 3a). The EDS analysis indicates the layer to be silica (SiO_2), which shows a semi-molten state with a few micron-size blind pores inside (Fig. 3b). It is believed that these pores were caused by the volatilization of SiO_2 at high temperatures during ablation [43]. The CS-2 sample was ablated under more severe environment than CS-1. Scraggy morphologies can be observed for CS-2 with more micron-sized pores in the formed SiO_2 layer (Fig. 3c). The SiO_2 layer shows a complete molten state (Fig. 3d) compared to that on the ablated CS-1 due to higher surface