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ZrB_2–SiC composite parts in oxyacetylenic torch tests: Experimental and computational assessment of chemical, thermal and mechanical behavior

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ABSTRACT

The thermal shock and ablative behavior of ZrB_2–SiC pyramidal mockups with different geometrical sizes were investigated using an oxyacetylene torch. Insignificant weight or configurational changes and the absence of surface cracks after testing were observed. The excellent resistance to thermal shock and ablation is attributed to the SiC addition. A satisfactory agreement between the measured temperature distributions and FEM computations was achieved. Results indicated that both the side length and radius of nose curvature exerted a prominent effect on the temperature gradient and thermal stress magnitude inside and/or on the surface of the pyramidal mockups. The magnitudes of heat flux and boundary layer temperature, T_e influenced the head temperature, T_1 and the undersurface temperature, T_2 in a different way. A stronger heat flux yielded higher T_1 value whereas T_2 remained almost constant. Comparatively, T_e showed a much more remarkable effect on T_2 than it did on T_1 owing to the different heat transfer mechanisms of these two parts.

1. Introduction

The thermal protection materials used in hypersonic aerospace vehicles and re-usable atmosphere re-entry vehicles must be able to withstand high heat flux and high temperature. Simultaneously, they must also be endowed with good oxidation, thermal shock resistance, ablation resistance and dimensional stability [1–4]. Among the ultra-high temperature materials (UHTMs), carbon fiber reinforced carbon matrix composite (C_f/C) has been considered as candidate for the aforementioned applications due to its low density, excellent high-temperature strength, high thermal conductivity and low coefficient of thermal expansion (CTE) [5–7]. Nevertheless, C/C composite exhibits quite poor oxidation and ablation resistance as it oxidizes above 500 °C. Although an oxidation-resistant coating may be effective to some extent in overcoming these drawbacks, cracking usually occurs during thermal cycling owing to the CTE mismatch between the C/C and coating systems [8]. As a result, the ultra-high temperature applications of C/C composite are undoubtedly restricted.

Transition metal borides including ZrB_2, HfB_2 and TiB_2, as another group of UHTMs, have attracted increasing attention and interest in recent years for their extremely high melting temperatures (ZrB_2 3040 °C, HfB_2 3250 °C), good thermo-chemical and thermo-mechanical properties, high emissivity and thermal conductivity [9–13]. Unfortunately, monolithic ZrB_2 exhibits limited sinterability due to its covalent bonding, high melting temperature, and low self-diffusion coefficient of Zr and B. Moreover, ZrB_2 is susceptible to catastrophic failure due to its inherent brittleness under thermal shock condition. Reported studies have indicated that SiC addition could enhance the densification and limit the grain growth of ZrB_2 efficiently [14,15]. Other invaluable findings demonstrate that ZrB_2–SiC composites show superior oxidation and thermal shock resistance as compared to monolithic ZrB_2 [16,17].

As major issues for thermal protection applications in severe oxidizing environments on hyposonic flight vehicles, thermal shock and ablation behavior of ZrB_2-based ultra-high temperature ceramics (UHTCs) have been investigated widely by many researchers. In general, there have been several heating approaches employed to carry out thermal shock and/or ablation tests, including oxyhydrogen torch [18], heating ovens [19,20], laser [5], plasma wind tunnel [21,22] and oxyacetylene torch [8,23]. The samples for most thermal shock experiments were heated to desired temperatures and then quenched in water or air. Under such a circumstance, the samples were subjected to a drastic temperature change for different thermal shock cycles usually resulting in thermal shock damage and/or catastrophic fracture. Several key parameters including residual strength, critical temperature difference, thermal stress and thermal shock parameters (R, R' and R'') have been examined to describe the thermal shock behavior [19]. In previous studies, samples with simple shapes, i.e. bars for thermal shock tests [18–20], flat-face, thin column or blunt hemisphere for ablation property

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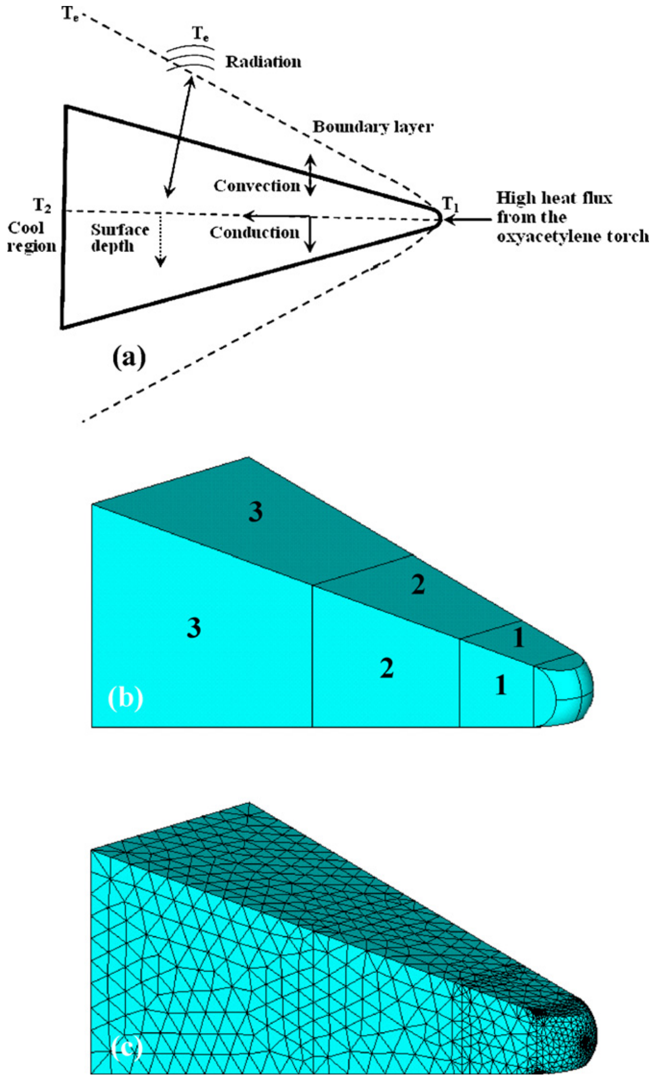

Fig. 1. (a) Surface energy balance where convection, conduction and radiation were involved, (b) outside contour of specimen divided into three parts, 1, 2 and 3 and (c) mesh for calculation by FEM of ZS-50-3 specimen.

assessment [5,8,21–23] were usually used. However, the design of high-performance hyposonic vehicles generally involves relatively sharp nose tips and wing leading edges (WLEs) with the purpose of reducing the vehicle's drag, enhancing maneuverability and performance, and improving the lift [21–24]. In this case, when the sharp nose or WLEs are heated, in particular by means of oxyacetylene torch, thermal shock due to the temperature gradient from sharp nose to empennage part will coexist with ablation and oxidation, if the atmosphere is oxidizing. Unfortunately, there have been quite limited reports on the shock/ablation behavior of ZrB_2–SiC with surface temperature higher than 2000 °C, much less on geometrical (shape and size) effects.

Therefore, the goal of this study was to perform tests on realistic parts made of ZrB_2–SiC, with an accurate control of the temperature distribution. The method was to introduce SiC particulates in ZrB_2 as oxidation inhibitors and densification accelerants for hot-pressed ZrB_2–SiC composite. The densified bulk composites were machined into sharp-nosed rectangular pyramid with different side length and radius of nose curvature radii. Their chemical, thermal, and mechanical behaviors were investigated through exposure to an oxyacetylene torch flame at ultra-high temperatures (∼2700 °C). The ablation and oxidation mechanisms were revealed via microstructural examination. The temperature and thermal stress distribution throughout the specimen were calculated by means of finite element method (FEM) analysis based on surface energy balance principle and a comparison was conducted with the experimentally measured results. The effects of specimen geometry, heat flux and boundary layer temperature were investigated.

Table 1
Geometry and abbreviated designation of rectangular pyramid specimens with rounded tips.

Abbreviated designation	ZS-30-3	ZS-50-3	ZS-70-3	ZS-50-1.5
Side length, *L* (mm)	30	50	70	50
Radius of nose curvature, *r* (mm)	3	3	3	1.5
No. of cells of the body mesh	23 798	21 918	17 334	8 422

2. Experimental procedures

2.1. Materials processing and sample preparation

Commercially available powders were used as raw materials in this study. The as-received ZrB_2 powders (Dandong Chemical Co., Ltd., Dandong, China) had a mass purity of >98.5% (with <1.5 mass% B_2O_3 as impurity) and an average particle size of ∼5 μm. SiC powders (a-phase, Shanghai Chemical Reagent Co., Shanghai, China) had a mass purity of >98.5% (impurity: <0.25% C and <0.5% Fe_2O_3) and an average particle size of ∼20 μm. The powder mixtures of ZrB_2–20 vol.% SiC (hereinbelow abbreviated as ZS) was ball-milled in ethanol and dried in heating oven at 80 °C. Milled powders were then uniaxially hot pressed in a BN-coated graphite die at 1850 °C for 120 min in vacuum under 30 MPa. The bulk density and theoretical density were evaluated using the Archimedes method and the rule of mixture, respectively. The densified compacts were machined by means of electric discharge machining into three types of samples for different tests. The bars with a size of 3 mm × 4 mm × 40 mm were used for Young's modulus evaluation using three-point bending test on a MTS 810 testing machine with a span of 30 mm and a crosshead speed of 0.5 mm/min. The flat shaped samples with a dimension of 33 mm × 30 mm × 3 mm were utilized for the measurement of heat flux from the oxyacetylene torch with different gas flow rates. In order to examine the effect of geometry on the chemical, thermal and mechanical behavior, several ceramic mockups with different side length, *L* and radius of nose curvature radii, *r* with an identical vertex angle of 18° were prepared by electric discharge machining (EDM). All the specimens were polished till the trace of wire cutting disappeared and Table 1 presented their abbreviated designations. For comparison, one mockup sample with $L = 30$ mm and $r = 3.0$ mm from ZrB_2–20 vol.% ZrO_2 (hereinbelow abbreviated as ZZ; ZrO_2 powders have a purity of 99.8% and an average particle size of 2 μm provided by Yixing Ceramic Co., Yiying, China) was also prepared using the same processing route as ZZ composite.

2.2. Thermal shock/ablation test and microstructural characterization

Thermal shock/ablation tests were performed in a flowing oxyacetylene torch environment. The oxyacetylene torch was obtained by a FeiHuan G01-30 cutting torch gun. Gas flows of O_2 and C_2H_2, measured by an LZB-10 glass rotameter were 0.34 m^3/h and 0.32 m^3/h, respectively. The distance between the nozzle tip of the oxyacetylene gun and the top of the specimen was 30 mm and the inner diameter of the tip was 0.7 mm. The heat flux of the torch was measured using flat-faced ZS sample with a dimension of thickness of 33 mm × 30 mm × 3 mm. The temperature to time (*T*–*t*) curves of the front surface and back surface temperatures of the sample