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Oxidation behavior in wet oxygen environment of Al_2O_3 added reaction-sintered Si-B-C ceramics

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ABSTRACT

The influence of Al_2O_3 addition on the oxidation behavior of Si-B-C ceramics at 1200 °C in O_2/H_2O (40/60) atmosphere was studied. Results showed that Al_2O_3 was enriched in the oxidized layer, and impeded the crystallization of oxide (cristobalite) during cooling. Denser oxidized layer and less weigh change were observed. Infrared spectra indicated that the addition of Al_2O_3 could weaken the tendency of bridged oxygen atoms (Si-O-Si, Si-O-Al) to become non-bridged oxygen atoms (Si-O-H) and enhance the degree of interconnection of the structural units. The above phenomenon was attributed to the fact that the bridging oxygen bond between Al and Si was not broken but protonated. The protonated bridging oxygen (Al-O(H)-Si) still acts as a linkage in the glass network, which results in higher viscosity of the aluminosilicates melt and lower reaction activity with steam, thereby significantly improving the oxidation resistance of Si-B-C in O_2/H_2O atmosphere.

1. Intruduction

SiC fiber-reinforced SiC composites (SiC_f/SiC) are considered as one type of the most promising engineering materials for thermal structural components in aero engine due to their excellent properties such as light weight, high strength, non-brittle mechanics, good reliability at high temperature and so on [1,2]. However, the matrix cracks resulted from the defects in preparation process and external stress limit the extensive use of SiC_f/SiC in high-temperature oxidizing environments [3]. To improve the oxidation resistance of SiC_f/SiC composites, boron-bearing materials, especially boron carbide (B_4C), are frequently introduced into the matrix. It is expected that the glass phase formed by the oxidation products of Si-B-C can seal the cracks under the action of capillary force and prevent the oxidizing medium diffusion into the inner interphase and fibers [4]. However, despite the excellent anti-oxidation and crack healing properties of SiC_f/Si-B-C composites, the susceptibility to corrosion of SiO_2 and the volatilization of B_2O_3 at high temperature (> 1100 °C) in water vapor environment weaken the self-healing ability and decrease the service life of SiC_f/Si-B-C composites [5,6]. Research by Liu [7,8] and Viricelle [9,10] all showed that volatilization of borosilicate glass would be accelerated when temperature exceeds 1000 °C in water vapor atmosphere. This could weaken the function/ability of borosilicate glass sealing cracks and lead to different degrees of damage to materials. It is necessary to improve the corrosion resistance of self-healing SiC_f/Si-B-C ceramic composite.

Researches on AlN-SiC-TiB_2 ceramics [11–13] oxidation behavior under air atmosphere showed that Al_2O_3 and B_2O_3 together will form aluminum borates ($Al_4B_2O_9$, Al_5BO_9) to limit the volatilization of B_2O_3. Furthermore, the effect of Al_2O_3 addition in BaO-Al_2O_3-B_2O_3-SiO_2 SOFC sealing glass on the improvement of B_2O_3 stability has also been reported [14]. Research by Cheng [15] showed that as the ratio of Al_2O_3/B_2O_3 increased in Al_2O_3–B_2O_3–SiO_2 glass system, the [BO_3] unit content decreased and the [BO_4] unit content increased because of the formation of Al-O-B bridging bond. In addition, Al_2O_3 is also a glass network intermediate as well as an important ingredient of environment barrier coating [16] (e.g. Mullite, BSAS). It can effectively improve the high temperature stability of silicate glass by repairing glass network structure [17]. Moreover, some researchers have indicated that Al_2O_3 could form a more complex group of aluminum silicon oxide anion ($Al_xSi_yO_z^{n-}$) with SiO_2 to improve melt viscosity [18]. Since the

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self-healing ability of the SiC_f/Si-B-C composites mainly comes from the Si-B-C matrix, it is very important to study the modification of the Si-B-C ceramic matrix. The above analysis showed that Al_2O_3 can not only form stable compounds with B_2O_3 to limit it volatilization, but also can form a more complex group ($Al_xSi_yO_z^{n-}$) with SiO_2 to improve melt viscosity. However, the research of modification of Si-B-C matrix with Al_2O_3 to improve its water vapor oxidation resistance is rarely reported to our knowledge. In this work, Al_2O_3 is introduced into Si-B-C ceramics as an oxidation product modifier for the improvement of the corrosion resistance of the materials in water vapor environment. Based on this research, it is expected that the modifier Si-B-C ceramic could be used as one kind of more corrosion resistant self-healing matrix of SiC_f/Si-B-C ceramic composite served in high temperature and H_2O/O_2 conditions.

2. Experimental procedure

2.1. Material preparation

SiC/B_4C powder mixture (molar ratio = 4:1) was ball milled together with different amounts of Al_2O_3 (wt% = 0, 5, 10) for 24 h, with ethanol as solvent and polyvinyl butyl as binder. After oven drying, the mixed powders were die-pressed into cylinder shaped samples (Φ 20 mm) at 30 MPa. Samples were pre-sintered for 2 h at 1600 °C in Ar atmosphere to get sufficient strength. The pre-sintered samples were impregnated with phenolic resin, and pyrolyzed at 900 °C for 1.5 h in Ar. Finally, the samples were infiltrated with melting silicon at 1500 °C under vacuum in a carbon tube furnace. The residual silicon is about 30%wt.

2.2. Oxidation test and microstructure characterization

Specimens of 5 mm × 4 mm × 3 mm in size were cut from the sintered plates with a diamond saw. Oxidation tests were performed in a tube furnace (GSL-1700X, Hefei Crystal Materials Technology Co., Ltd., Hefei, China) under normal pressure with flowing O_2/H_2O atmosphere at 1200 °C up to 98 h. During the test, the specimens were supported on an Al_2O_3 crucible, and the flow rate of O_2/H_2O was kept constant at 500 ml/min (200 ml O_2 + 300 ml H_2O) by a liquid phase vaporization system (LVD-F1, Hefei Crystal Materials Technology Co., Ltd., Hefei, China) (Fig. 1). The heating and cooling rates were 8 °C/min and 5 °C/min respectively. The weight changes of specimens before and after oxidation were recorded, using an analytical balance (MT5, Mettler Toledo Co., Ltd., Switzerland) with an accuracy of ± 0.001 mg. The internal vibration modes and microstructures of the oxide glasses were studied using Micro zone-Raman spectroscopy and Infrared spectra. The NMR spectra were used to detect the peripheral molecular environment of ^1H and ^{27}Al. The surface and cross-sections of the oxidized specimens were characterized using a S4800 field emission scanning electron microscope (Hitachi, Tokyo, Japan) along with energy-dispersive spectroscopy (EDS, Inca energy) for elemental analysis.

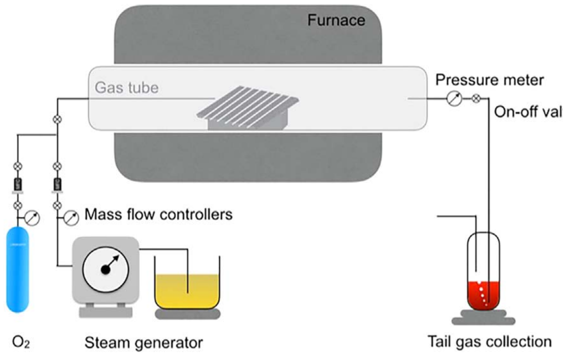

Fig. 1. Experimental device schematic diagrams.

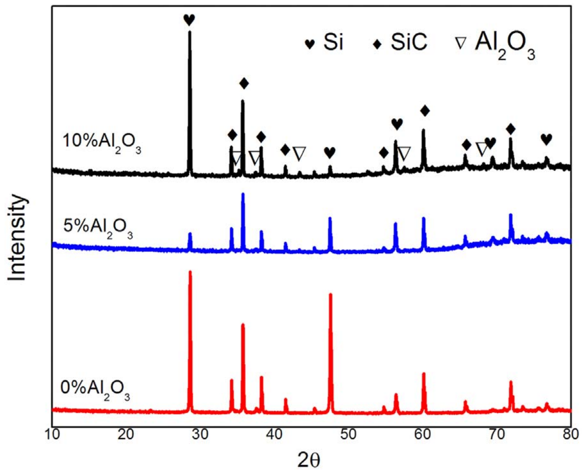

Fig. 2. XRD spectra of reaction sintered Si-B-C ceramics with different Al_2O_3 contents.

3. Results

3.1. Microstructure of the as-processed samples

XRD patterns of the reaction-sintered Si-B-C ceramics doped with various amounts of Al_2O_3 were shown in Fig. 2. All samples show distinctive peaks of SiC and Si. The diffraction peaks of B_4C are not found, which can be attributed to the diffusion of Si atom into B_4C during reaction-sintered [19]. In addition, diffraction peaks of Al_2O_3 gradually appear with the increase of Al_2O_3 content. The diffraction peaks of Al_2O_3 are obviously observed in sample with 10% Al_2O_3. B_4C, SiC, Si and Al_2O_3 are homogeneously distributed as shown in Fig. 3.

3.2. Oxidation behavior

The oxidation behaviors of the samples under different oxidation atmosphere are analyzed according to the weight changes. Fig. 4 shows the weight gain of the reaction-sintered Si-B-C ceramics after oxidation in different atmosphere (Fig. 4(a): dry O_2, Fig. 4(b): O_2/H_2O) for same period at 1200 °C. The addition of Al_2O_3 plays an important role in affecting the weight gain. Independent of the oxidation atmosphere, sample with Al_2O_3 always shows lower weight gains compared with the samples without Al_2O_3, and the weight gain difference becomes increasingly large with the prolongation of oxidation time. The above results could be attributed to that the addition of Al_2O_3 can effectively increase the viscosity of oxidized phase and improve the resistance of oxygen diffusion [18]. Under dry O_2 atmosphere, the oxidation curves are parabolas-like, which indicates that the oxidation is controlled by the diffusion of oxygen through the oxidation layer [20]. While under the O_2/H_2O atmosphere, their oxidation curves slope increase rapidly, indicating that under the corrosion of O_2/H_2O, the decrease of viscosity in the oxidation layer makes it easier for oxidizing medium to diffuse into the interior of the material [21]. However, as Al_2O_3 increases, the oxidation curve slope gradually reduces, explaining that the viscosity of the oxidation layer is still large. Such a phenomenon indicates that the oxidation of the material under water vapor is significantly enhanced and the addition of Al_2O_3 can improve the anti-oxidation ability of reaction-sintered Si-B-C ceramics in O_2/H_2O atmosphere. However it's necessary to point out that the weight gain is the result of two simultaneous processes that are oxidation weight gain and volatilization weight loss. Therefore, the appeared weight gain is not solid to perfectly reflect the anti-oxidation behavior.

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