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Oxidation behavior of C/SiC-Ti_3SiC_2 at 800–1300 °C in air

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ABSTRACT

In this work, the oxidation behavior of C/SiC-Ti_3SiC_2 from 800 to 1300 °C has been studied compared with that of C/SiC. C/SiC-Ti_3SiC_2 shows severe weight loss and low flexural strength after oxidation of 10 h at 800 °C. With the oxidation temperature increasing to 1000 °C, the coating cracks can be healed by silica and the matrix cracks can be healed by the oxidation product of Ti_3SiC_2, so the strength retention ratios of C/SiC-Ti_3SiC_2 reach to above 90% after oxidation of 10 h at 1000 and 1200 °C, revealing better oxidation resistance than C/SiC at the temperature range from 1000 to 1200 °C. With the oxidation temperature increasing to 1300 °C, the flexural strength decreases after oxidation of 10 h due to the oxidation of outer carbon fibers.

1. Introduction

Carbon fiber reinforced SiC matrix composite (C/SiC) has been considered as an important high-temperature structural material due to the low density, remarkable mechanical properties and excellent thermal stability [1–3]. In order to extend the application field of C/SiC, various researches have been carried out to introduce second phase into C/SiC to improve the performance. Up to now, the researches focus on two aspects, the first one is to introduce the B-containing phases to improve the oxidation resistance at low temperatures [4–7], and the second one is to introduce refractory phases to improve the ablation resistance [8–10]. In recent years, MAX phases join the group of improving the performance of C/SiC.

MAX phases, as a new group of advanced ceramics, own the combined properties of ceramics and metals due to the special atomic bonding and laminated structure [11–13]. In order to introduce MAX phases into C/SiC, different methods have been tried. Lenz et al. fabricated the composites by the combination of polymer infiltration and pyrolysis (PIP) and liquid silicon infiltration (LSI), and the TiC and Al powder were added into phenolic resin to react with silicon melt to form MAX phases in the matrix [14]. Yang et al. fabricated the composites by PIP, in which Ti_3SiC_2 powder was added into polycarbosilane (PCS) as inert filler [15]. In our previous works, the combined process of slurry infiltration and LSI has been applied to in situ form MAX phases into C/C-SiC and C/SiC, and the composites reveal improved tribological performance, ablation resistance, mechanical and electromagnetic interference shielding properties [16–20]. Additionally, the similar works also can be found on the introduction of MAX phases into SiC/SiC. Spencer et al. tried to fabricate SiC fiber reinforced Ti_3SiC_2 matrix composites by hot pressing [21]. Mu et al. prepared SiC/SiC containing Ti_3SiC_2 by PIP, and the composites show excellent mechanical and dielectric properties [22]. Up to now, the oxidation resistance of ceramic matrix composites (CMC) containing MAX phases still cannot be found. Oxidation resistance is important for the application of structural materials at high temperatures, and it is essential to study the oxidation resistance of CMCs containing MAX phases.

MAX phases, especially the Al-containing MAX phases, Ti_3AlC_2 and Ti_2AlC, reveal good crack-healing properties due to the formation of a stable and well-adhering Al_2O_3, which can recover the parent matrix strength resulting from the high mechanical properties of Al_2O_3 close to matrix strength [23–26]. Up to now, the research on the crack-healing effect of Ti_3SiC_2 is rarely seen. The volume expansion can happen during the oxidation of Ti_3SiC_2, and the formed TiO_2 and SiO_2 can fill the cracks but cannot well recover the parent matrix strength due to the low strength of TiO_2 and SiO_2. However, for the improvement of oxidation behavior of C/SiC, it requires the crack-healing effect without the requirement of bonding strength. Thus, it is possible to improve the oxidation resistance of C/SiC by the introduction of Ti_3SiC_2.

In our previous works, Ti_3SiC_2 has been introduced into C/SiC by densification process of LSI [18,19]. The composite has a low porosity, and the existence of crack-healing phase is possible to heal the cracks in the oxidation process, both of which are beneficial to improving the oxidation resistance. In this work, the oxidation behavior of C/SiC-Ti_3SiC_2 in air at the temperature range from 800 to 1300 °C was investigated, and the microstructure evolution in the oxidation process was revealed.

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2. Experimental

2.1. Materials preparation

Two-dimensional carbon fiber preforms (1K, T-300^{TM}, Toray Co., Japan) with a fiber volume fraction of 40 vol.% were employed. PyC interphase with a thickness of 200 nm was deposited, and then the preform was heat treated at 1800 °C for 2 h under vacuum. Methyltrichlorosilane (MTS, CH_3-$SiCl_3$) was used for the deposition of SiC matrix. MTS vapor was carried by bubbling hydrogen. The conditions for deposition of SiC matrix were as follows: the deposition temperature was 1000 °C, pressure was 5 kPa, time was 240 h, and the molar ratio of H_2 to MTS was 10. After that, the porous C/SiC preform can be obtained.

The porous C/SiC preform was infiltrated with TiC-C slurry (60 wt.% TiC, 6 wt.% graphite), and then the preform was infiltrated with liquid silicon at 1600 °C for 30 min under vacuum. After that, the as-received composites were machined into the samples with dimensions of $40 \times 5 \times 3\ mm^3$. The outer carbon fibers would be naked after machining, hence two layers of SiC coating were deposited on the machined samples to protect the naked fibers, and C/SiC-Ti_3SiC_2 with density of 2.42 g/cm^3 and open porosity of 8 vol.% was obtained.

The porous C/SiC preform continued to be infiltrated by SiC matrix for 240 h, and then was machined into the samples with dimensions of $40 \times 5 \times 3\ mm^3$. Finally, two layers of SiC coating were deposited on the machined samples, and C/SiC with density of 2.0 g/cm^3 and open porosity of 14 vol.% was obtained.

2.2. Oxidation tests

The oxidation tests were conducted in static air in an alumina tube furnace at 800, 1000, 1200 and 1300 °C. The furnace was firstly heated to the given temperature, and then the samples were directly pushed into the furnace. The weights of specimens were measured by an analytic balance (sensitivity: 0.01 mg, Mettler AG 105) after each oxidation for 1, 3, 5, 7 and 10 h at the given temperature, and then the oxidation weight change $\Delta m/m_0 = (m - m_0)/m_0$, where m_0 and m are the weight of samples before and after oxidation, can be calculated. Five specimens were measured in each case for statistical significance.

2.3. Characterization

The flexural strength of samples with dimensions of $40 \times 5 \times 3\ mm^3$ was tested by three-point bending method (SANS CMT 4304, Sans Materials Testing Co., Ltd., Shenzhen, China). The loading rate was 0.5 mm/min and the outer span was 30 mm. The coefficient of thermal expansions (CTE) of samples with dimensions of $25 \times 3 \times 3\ mm^3$ were tested by DIL 402C (NETZSCH, Germany). The measurements were conducted from RT to 1400 °C in argon atmosphere with a heating rate of 5 °C/min. The microstructure of samples was characterized by scanning electron microscope (SEM, S-2700, Hitachi, Japan) and back-scattered electron image (BSE). Energy dispersive X-ray spectrum (EDS) was recorded to identify the element species.

3. Results and discussion

3.1. Microstructure

The microstructures of C/SiC and C/SiC-Ti_3SiC_2 are shown in Fig. 1. Due to the bottle-neck effect, both intra-bundle and inter-bundle pores exist for C/SiC (Fig. 1a). For C/SiC-Ti_3SiC_2, the inter-bundle pores are filled by the in-situ formed matrix in LSI process (Fig. 1b), leading to the denser inter-bundle matrix and the lower porosity than C/SiC. A homogeneous and dense SiC coating with a thickness of about 100 μm can be found for these two composites. As shown in Fig. 1a and b, it can be found that the matrix cracks exist resulting from the release of thermal residual stress (TRS) during the cooling down from fabrication temperature to room temperature due to the CTE mismatch between carbon fiber and matrix. At the same time, the cracks also exist in SiC coating due to the CTE mismatch between the composites and SiC coating, as shown in Fig. 1c.

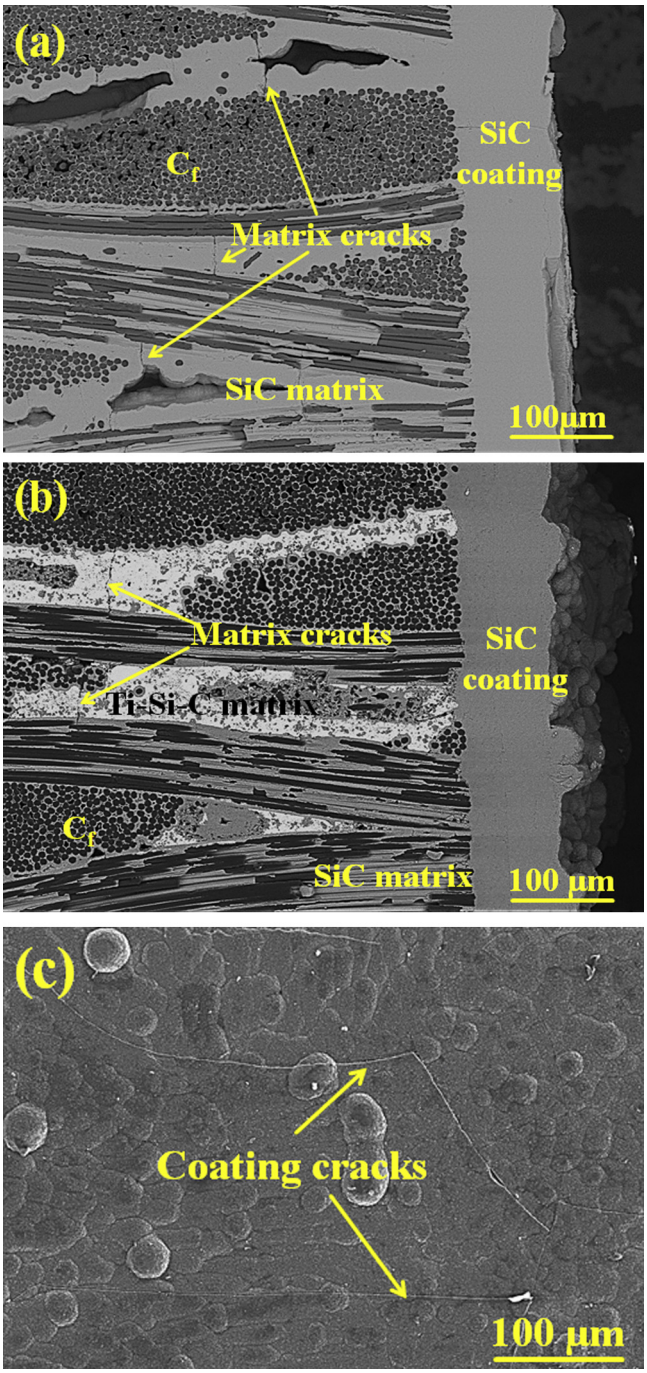

Fig. 1. Microstructure of (a) C/SiC, (b) C/SiC-Ti_3SiC_2 and (c) SiC coating.

3.2. Weight variation

As shown in Fig. 2a, with the increase of oxidation time, the weight loss of C/SiC increases linearly for the oxidation at