

# **Oxidation behavior of 3D-C/SiC composites fabricated by PIP process at low temperatures**

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**Abstract.** The oxidation behavior of C/SiC composites, prepared at low temperature as 900°, comparatively lower than currently reported fabrication temperatures, was investigated, which indicated that the C/SiC composites started to oxidize at about 600°C, and the flexural strength was enhanced diversely after oxidated under 800°C, and then reduced in succession with the oxidative temperature increasing. In particular, the flexural strength reached 701.46MPa after oxidated at 600°C. The vitreous SiO<sub>2</sub>, oxidative product of C/SiC composites, maybe protected the reinforced carbon fibers and remedied the holes and crackles, and then consequently enhanced the performance of C/SiC composites.

## **Introduction**

C/SiC composites, in possession of excellent properties of low density, high strength and oxidation resistance, have been used in a lot of fields, especially in aeronautics and astronautics, such as thrust chamber in rocket engines and thermal protection system in re-entry spacecrafts [1-3]. As structural materials for high temperature applications in atmosphere, the C/SiC composites must have excellent oxidation resistance [4-6]. In the literature [7, 8], the mechanical properties and microstructure of C/SiC composites fabricated at low temperatures was investigated in depth, and the results showed that the C/SiC composite possessed outstanding performance. In order to adapt for application in oxidation environment as atmosphere, it is essential to study the oxidation resistance of C/SiC composites prepared at low temperatures.

In this paper, the oxidation behavior of C/SiC composites, fabricated at low temperatures, was investigated, and the oxidative mechanism was discussed too.

## **Experiment**

C/SiC composites were prepared by PIP process at low temperature as 900°C. PCS, the precursor of SiC matrix, with number-average molecular weight about 1800 and soften point about 205°C, was synthesized in National University of Defence Technology. The carbon fibers (Jilin Carbon Corp., China), with tensile strength and elastic modulus of 4.24GPa and 210GPa respectively, were firstly heat treated at 600°C for two hours in vacuum before PIP process. In succession, the carbon fiber preforms were fixed in a graphite mold, infiltrated with PCS solution in vacuum, and then pyrolyzed at 900°C for about 12 repetitions of PIP process for densification.

The density of C/SiC composites was measured using Archimedes method with kerosene as medium. The flexural strength was tested on a universal machine (WDW-100), and five specimens were measured for each sample. The C/SiC composites specimens were oxidized at designated temperatures for 10 minutes in muffle furnace, and then analyzed by scanning electron microscopy (SEM, JSM-5600LV).

## Results and discussion

The oxidation of C/SiC composites is a complicated reaction, which doesn't only include the weight loss due to the oxidation of carbon fibers and excess carbon in pyrolyzate of PCS, but also the weight increasing owing to the SiC matrix, just shown in equation (1) to (4).



Of course, the complexity of the oxidation of C/SiC composites isn't limited to the reaction of substrate, and it is an important aspect that there are lots of holes, crackles and complicated structure in the C/SiC composites. And the latter brings the oxidative characteristics of C/SiC composites, especially for the composites prepared at low temperatures.

Fig. 1 shows the remaining mass-oxidative temperature curves of C/SiC composites prepared at 900°C, and the heat preservation at designated temperature is 10 min. As is shown that the C/SiC composite, prepared at 900°C, starts to oxidize at about 600°C, and the weight loses quickly after 700°C, which is consistent with what the literature [9] described.

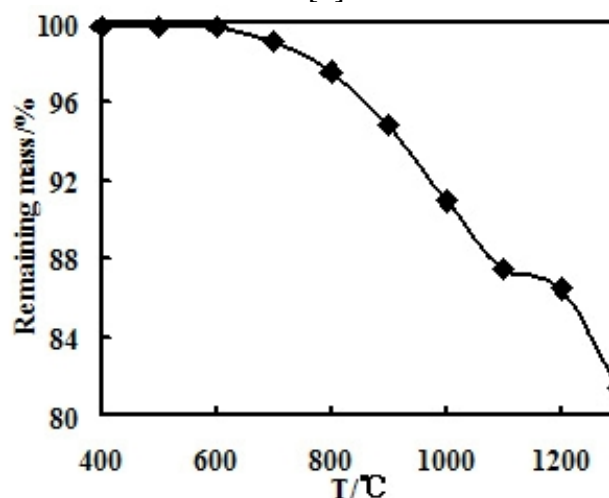


Fig. 1 Flexural strength-oxidated temperature curves for C/SiC composites

In comparison with the weight loss, it is more complicated that the flexural strength of C/SiC composites, prepared at low temperatures, changes with the oxidative temperature increasing. Just as shown in Fig. 2, the flexural strength of C/SiC composites, fabricated at 900°C, are enhanced diversely after oxidated under 800°C, compared with the original flexural strength 542.13MPa. In particular, the flexural strength of C/SiC composites, oxidated at 600°C for 10min, reaches 701.46MPa, remarkably higher than the property of C/SiC composites reported. And then the flexural strength reduces in succession with the oxidative temperature increasing just like described in literature [9]. This is different from other C/SiC composites.

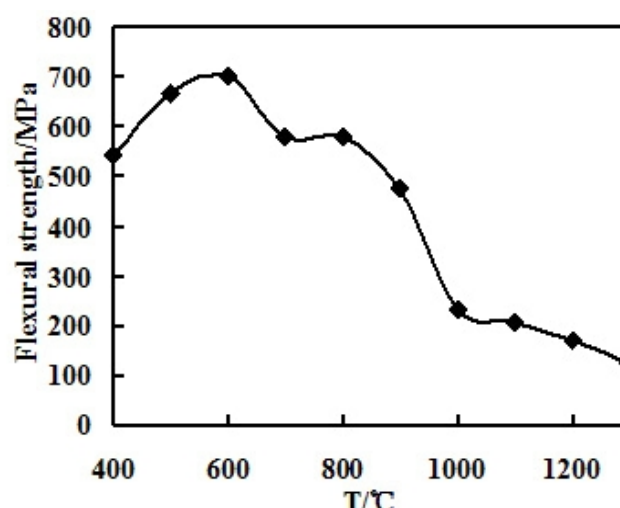


Fig. 2 flexural strength-oxidated temperature curves for C/SiC composites

In order to explore why the low temperature oxidative characteristics of C/SiC composites, prepared at low temperatures, differ from other C/SiC composites, the surface of C/SiC composites was tested by SEM, just as shown in Fig. 3. We can see from Fig. 3A that the surface of C/SiC composites after oxidated at 600°C doesn't only have matrix, but also the bare carbon fibers. There are some obvious holes, crackles on the surface of the bare carbon fibers on account of oxidation, just as shown in Fig. 3B. The oxidation of carbon fibers, the reinforced fibers in C/SiC composites, will badly depress the properties of C/SiC composites, so the protection of carbon fibers in C/SiC composites maybe keeps the performance of C/SiC composites. After oxidated, the matrix of C/SiC composites will turn into vitreous body, which can fold the bare carbon fibers and fill up the holes and crackles in the C/SiC composites like flow water just as shown in Fig. 3A, Fig. 3C and Fig. 3D. In particular, the latter must be responsible for the remarkable properties of C/SiC composites after oxidated. Furthermore we can see obvious flowing trace like liquid from Fig. 3D.

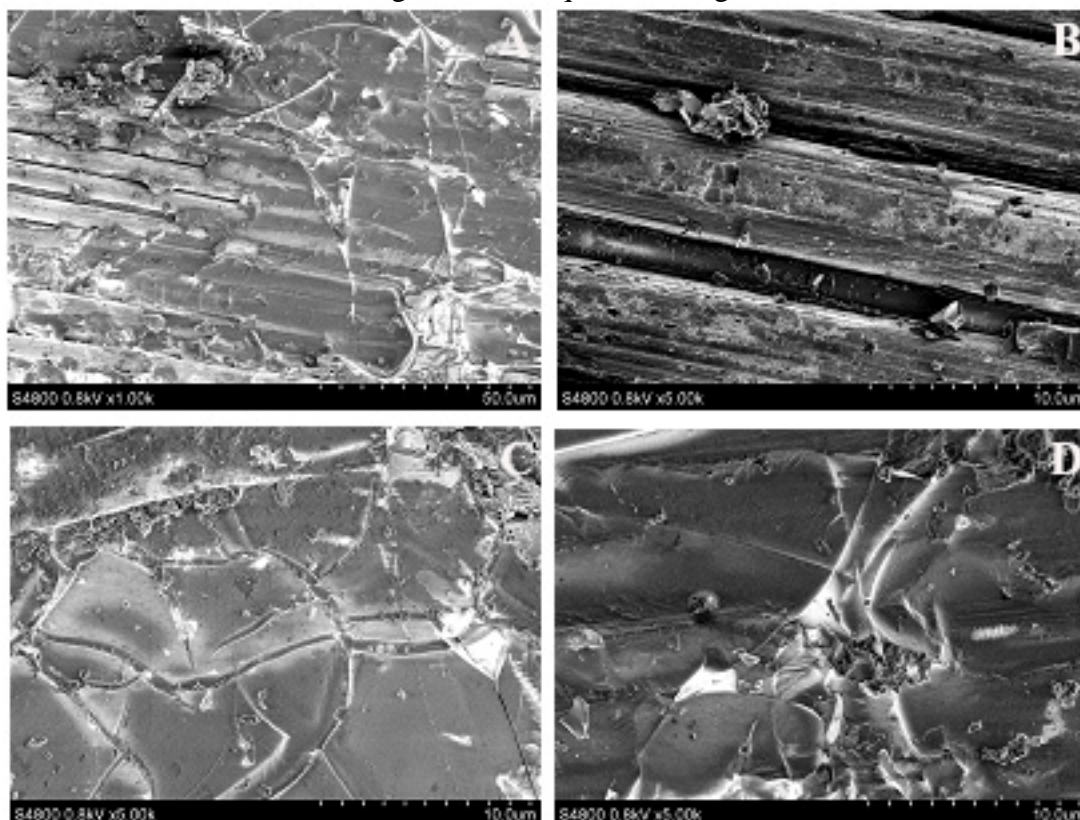


Fig. 3 The surface of C/SiC composites after oxidated at 600°C

Fig. 4 is the EDAX of vitreous body on the surface of C/SiC composites after oxidated at 600°C. The results show that the vitreous body is composed of element C, O and Si. The atom percent of Si and O is 17.83% and 32.58% respectively, and the proportion is 1.09:2, just like the ratio of SiO<sub>2</sub>. In virtue of the presence of vitreous SiO<sub>2</sub>, which doesn't only protect the reinforced carbon fibers, but also remedy the holes and crackles in the surface of C/SiC composites, the flexural strength of C/SiC composites are enhanced consequently. Of course, the element C should be the carbon fibers or the excess carbon in pyrolyzate of PCS.

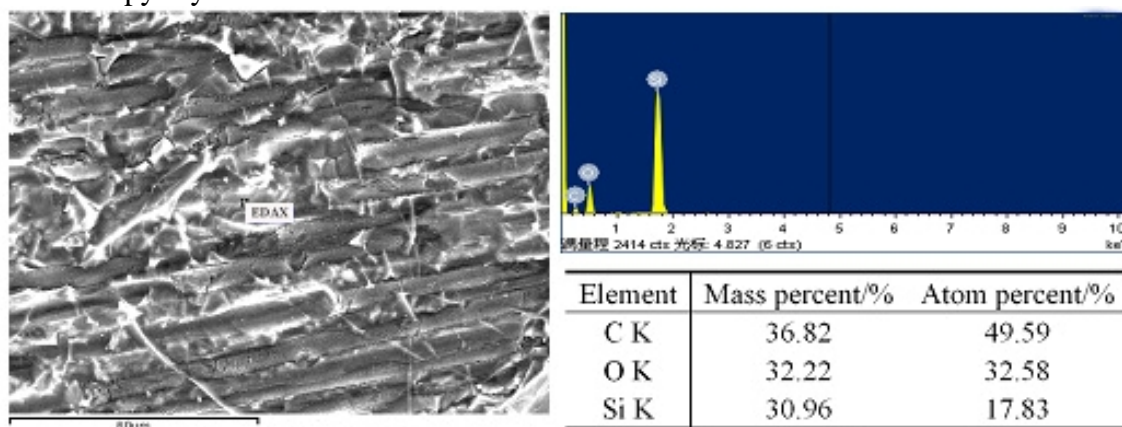


Fig. 4 The EDAX of vitreous body on the surface of C/SiC composites after oxidated at 600°C

## Conclusions

The oxidative damage of reinforced carbon fibers is the primary matter which depresses the performance of C/SiC composites. The C/SiC composite, prepared at 900°C, starts to oxidize at about 600°C, and the weight loses quickly after 700°C. It is distinguished from other composites that the flexural strength of C/SiC composites is enhanced to higher than its original property after oxidated under 800°C for 10 min. Especially after oxidated at 600°C, the flexural strength reaches 701.46MPa, while its original property is just 542.13MPa. The vitreous SiO<sub>2</sub>, oxidative product of C/SiC composites, maybe protects the bare carbon fibers and remedies the holes and crackles, and then enhances the microstructure and performance of C/SiC composites.

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