Environmental Performance Test and Analysis of SiC/SiC Composites

Mingwei Chen\textsuperscript{1,2}, Haipeng Qiu\textsuperscript{1,2}, Weijie Xie\textsuperscript{1,2}, Bingyu Zhang\textsuperscript{1,2}

\textsuperscript{1} AVIC Composite Technology Center, Beijing, China, 101300
\textsuperscript{2} AVIC Composite Co., Ltd., Beijing, China, 101300

E-mail: mingwei070806@163.com

Abstract: SiC/SiC ceramic matrix composites were prepared by polymer impregnation pyrolysis (PIP) process with domestic first-generation SiC fibers as reinforced phase and SiC ceramic as matrix phase. The structure and morphology of SiC ceramic matrix and SiC/SiC composites were investigated by means of XRD and SEM method. Furthermore, the mechanical properties of SiC/SiC composites after high temperature gas testing were investigated by flexural strength using three point bending testing. Results showed SiC/SiC composites have excellent mechanical properties at room temperature with bending strength of 330 MPa. However, obvious cracks and serious oxidation appeared after up to 150 hours of high temperature gas testing, and the mechanical properties dropped dramatically with the flexural strength retention rate less than 50%. The decrease of the mechanical properties and the occurrence of cracks of SiC/SiC composites were mainly attributed to the structure evolution and the oxidation intensification which might lead to instability of material properties and component structure.

1. Introduction
With the increase of thrust-to-weight ratio of aero engine, hot section components will suffer harsh heat flux and aerodynamic loads. So, excellent environmental properties, especially in high temperature oxidation and gas environment, are required to meet the need of high thrust-to-weight ratio aero engine. According to the research reports, the aero engine turbine inlet temperature increases substantially to 1500$^\circ$C and 1800$^\circ$C when the thrust-to-weight ratio reaches 10 and among 12-15, which poses strong challenges to the hot section components materials. The service temperature for conventional superalloy materials such as nickel alloys is about 1000$^\circ$C, which is difficult to meet the development requirements of hot section components for high thrust-to-weight ratio aero engines. Therefore, the research and development of high performance material with light weight, high temperature resistance, oxidation resistance, high toughness and long lifetime has become the most promising and feasible way, which have received extensive attention and input for many scientific research institutions and manufacturers in many countries and regions [1-3].

SiC/SiC composites are typical anisotropic materials with continuous reinforced SiC fiber as dispersed phase and SiC ceramic matrix as continuous phase. Therefore, SiC/SiC composites have the advantages of high temperature resistance, oxidation resistance, wear resistance and corrosion resistance of ceramic materials. Meanwhile, SiC/SiC composites overcome the inherent disadvantages of low fracture toughness and poor resistance to external impact load of traditional ceramic materials.
due to the strengthening and toughening effect of ceramic fibers. The special composition and structural characteristics mentioned above make SiC/SiC composites pose the advantages of light weight, high temperature resistance, oxidation resistance and excellent mechanical properties, which would replace the application of superalloys in hot section components of aero engines and was expected to become the promising structural material in the fields of spacecraft and nuclear technology [4-8].

In this paper, SiC/SiC ceramic matrix composites were obtained through polymer impregnation pyrolysis (PIP) process with domestic first-generation SiC fibers as reinforced phase and SiC ceramic as matrix phase. The structure and morphology of SiC ceramic matrix and SiC/SiC ceramic matrix composites were investigated by means of XRD and SEM methods. Furthermore, the high temperature failure mechanism of SiC/SiC composites was also explored, which would provide theoretical and technical support for environmental testing and installation assessment of SiC/SiC composite components in the future.

2. Experimental procedure

2.1. Preparation of SiC/SiC composites
SiC/SiC ceramic matrix composites were obtained through polymer impregnation pyrolysis (PIP) process. The main preparation steps included vacuum impregnation, pyrolysis and hot molding.

2.2. High temperature gas environment test of SiC/SiC composites
SiC ceramic was placed in a muffle furnace from room temperature to 1400°C. SiC/SiC composites were placed on a high temperature gas testing platform at about 1200°C. Then the SiC ceramic and SiC/SiC composites were taken out for further designed testing.

2.3. Detection and characterization
The phase structure and of SiC ceramic and SiC/SiC composites was analyzed by XRD on PANalytical X’Pert Pro X-ray diffractometer. The morphology of SiC/SiC composites was characterized by SEM on Hitachi S-4800 field emission scanning electron microscopy. The mechanical properties of SiC/SiC composites were characterized by three point bending method in the universal material testing machine (WDW-5S) based on GB/T 6569-2006.

3. Results and discussion

3.1. Micromorphology analysis of SiC/SiC composites
Cracks on the surface of SiC/SiC composites appeared after high temperature gas testing for about 150 hours at 1200°C. SiC/SiC composites were divided into seven parts according to the distance from the crack shown in Figure 1, wherein zone 5 was a crack region, zones among 1 to 4 were near crack regions, and zone 6 and zone 7 were far from crack regions. Then, the micromorphology of the seven regions were observed by SEM method.

For the crack zone 5, three test points were designed shown in Figure 2. Zone 5-1, Zone 5-2 and Zone 5-3 were respectively set at the outermost side, middle zone and innermost side of the crack zone. Then, the micromorphology of Zone 5-1, Zone 5-2 and Zone 5-3 mentioned above were tested by SEM shown in Figure 3, Figure 4 and Figure 5. Most pullout fibers pull out and breakage were found successively and gradually from the SiC ceramic matrix under high temperature impact load with the crack of about 0.50mm at the outermost surface of the crack shown in Figure 3(Zone 5-1), which would support typical energy absorption mechanisms to achieve the reinforcement and toughening effect of the SiC/SiC composites. According to the middle zone(Zone 5-2) of the crack shown in Figure 4, the crack width was reduced to 0.1~0.2mm, and SiC fibers were pulled out of the matrix but had not yet completely broken under continuous impact load, which further verified the energy absorption mechanisms under the impact of high temperature gas environment. The micromorphology
of the outermost side of the crack zone (Zone 5-3) shown that a small amount of thermal protective coating peels off which might cause the SiC fiber to be partially exposed to the high temperature gas atmosphere. SiC/SiC composites could maintain structural stability, but the original material structure might change and mechanical properties would be degraded under the continuous high temperature gas load and heat flow impact.

According to the surface and cross section micromorphology of the three test points, SiC/SiC composites posed typical feature of pseudoplastic fracture behavior with a large number of fibers debonding, pullout and breakage from SiC matrix. Furthermore, the section of the detection point zone 5-1 was relatively flat relative to other testing points. Relatively the most pullout fibers pullout were found and SiC fibers were pulled out successively and gradually from the SiC ceramic matrix, which would support typical energy absorption mechanisms more importantly for zone 5-3.

![Figure 1](image1.png)

**Figure 1.** Photo of SiC/SiC composites after high temperature gas testing.

![Figure 2](image2.png)

**Figure 2.** Different detection positions for crack region (zone 5).

![Figure 3](image3.png)

**Figure 3.** Micromorphology of the middle zone of the crack zone (Zone 5-1).
Figure 4. Micromorphology of the innermost side of the crack zone (Zone 5-2).

Figure 5. Micromorphology of the outermost side of the crack zone (Zone 5-3).

Figure 6. Micromorphology of the near crack regions (zone 1).

Micromorphology of four zones near crack regions named zone 1, zone 2, zone 3 and zone 4 were shown in Figure 6, Figure 7, Figure 8 and Figure 9 respectively. The micromorphology of two zones far from crack regions named zone 6 and zone 7 were also shown in Figure 10 and Figure 11. Moreover, it should be noted that the exfoliation area in the figure was formed during the testing samples preparation process instead of the morphology of the SiC/SiC composites after high temperature gas testing. Results showed that SiC/SiC composites maintained excellent structural
stability with uniform and compact thermal protective coating and only a small amount of cracks of the coating were found for near crack zones among zone 1 to zone 4 and far crack zones in zone 6 and zone 7. The testing results mentioned showed the structural characteristics of the thermal protective coating after the high temperature gas testing help to protect the stability of and inhibit the oxidation reaction of SiC/SiC composites.

Figure 7. Micromorphology of the near crack regions (zone 2).

Figure 8. Micromorphology of the near crack regions (zone 3).

Figure 9. Micromorphology of the near crack regions (zone 4).
3.2. Composition and phase analysis of SiC/SiC composites

Three points of SiC fiber at the outermost side of the crack (zone 5-1) are randomly selected for elemental analysis and the test results were shown in Figure 12. Results showed that the outermost crack was oxidized seriously with the average oxygen content as high as 36%. Moreover, obvious crystal structure differences were found at 22.15° which meant the characteristic diffraction spectra of SiO₂ at 800°C. When oxidation temperature increased to 1200°C and 1400°C, the diffraction peaks became much sharper which meant the formation of higher crystallization of SiO₂ (Figure 13), which meant an increase of the oxidation reaction.

3.3. Mechanical properties of SiC/SiC composites

The flexural strength at zone 1, zone 2, zone 3 and zone 4 was 110.00MPa, 70.15MPa, 74.75MPa and 119.58MPa respectively. Moreover, the room temperature strength of SiC/SiC composites reached 330MPa, which meant all the flexural strength retention rate of testing samples after high temperature gas testing was less than 40%. The significant decline in mechanical properties was mainly due to the motion interference between SiC/SiC composites and test fixture during the high temperature gas testing, which might cause the partial damage to SiC/SiC composites, especially to the thermal protective coating.
Furthermore, the stress-strain curves were also obtained to explain the fracture behavior of materials shown in Figure 14-17. All the fracture behaviors of SiC/SiC composites at different zones exhibited similarly. That was, the loading increased in a nonlinear dynamic path in the initial stage of loading. After reaching the maximum, the load remained basically unchanged for a wide displacement range, and then decreased in a ladder-like trend rather than dropped rapidly.
Figure 14. Stress-strain curves for zone 1 of SiC/SiC composites on zone 1.

Figure 15. Stress-strain curves for zone 1 of SiC/SiC composites on zone 2.

Figure 16. Stress-strain curves for zone 1 of SiC/SiC composites on zone 3.
Figur 17. Stress-strain curves for zone1 of SiC/SiC composites on zone 4.

In short, SiC/SiC composites possessed excellent pseudo-elastic behavior, although the mechanical properties dropped rapidly after high temperature gas testing, which will be favorable to increase the strength and toughness in the future.

4. Conclusion
(1) SiC/SiC ceramic matrix composites were prepared by polymer impregnation pyrolysis (PIP) process, and then high temperature gas testing were carried out at about 1200°C for 150 hours.
(2) The main reason for the above situation was that the motion interference between SiC/SiC composites material and test fixture during the high temperature gas testing, which might cause the partial damage to SiC/SiC composites, especially to the thermal protective coating.
(3) SiC/SiC composites posed typical pseudoplastic fracture characteristics of ceramic matrix composites after high temperature gas testing.

References
[1] Evans A G, Marshall D B 1989 Acta Materialia 37 2567
[2] Chen M W, Qiu H P, Jiao J 2013 Key Engineering Materials 544 43
[3] Naslain R 2004 Composites Sci Technology 64 155
[4] DiCarlo J A, Yun H M, Hurst J B 2004 Applied Mathematics and Computation 152 473
[5] Udayakumar A, Ganesh A S, Raja S, Balasubramanian M 2011 Journal of the European Ceramic Society 31 1145
[6] Jian K, Chen Z H, Ma Q S 2007 Ceramics International 33 73
[7] Hilling W 1994 Am. Ceram. Soc. Bull. 73 56
[8] Yamada R, Taguchi T, Igawa N 2000 J Nucl Mater 283–287 574