Study on the Performance of an Ultra High Temperature Ceramic Material

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ABSTRACT: With the development of technology and the requirements, the requirement on materials performance is more and higher in all walks of life. Especially in aerospace and other high-tech industries is more demanding materials with high temperature resistance, erosion resistance and other characteristics. Niobium alloy currently used in combustion chamber and Cf/SiC ceramic matrix composites has been unable to meet the requirements of the new generation of aircraft. It studied the preparation conditions and performance of Cf/ZrC-SiC high temperature resistant ceramic matrix composite materials. It founds that the infiltration time is 360min, the infiltration temperature is 1700℃, the samples in mechanical properties, thermal physical properties, anti erosion performance is very excellent.
KEYWORD: High temperature resistance; Ablation resistance; Ceramic matrix composite

1 BACKGROUND

At present, the high-tech development is very rapid, and the demand for materials is increasing. Especially, the application of aerospace field as the representative, they require the high temperature resistance, ablation resistance, high strength, high stiffness and high toughness etc. They also put forward higher requirements on the performance of the engine combustion chamber material. The material has high temperature resistance, high strength, low density and micro ablation characteristics (Zhang Xinghong et al, 2015). The niobium alloy combustion chamber and Cf/SiC ceramic matrix composites combustion chamber cannot be more than 1800℃ environment used for a long time. It has been unable to meet the needs of a new generation of aircraft development, so the development of new high temperature resistant material is imminent.

Ultra-high temperature ceramics is ZrB2, TaC, HfB, HfB 2, ZrC and other high-melting (above 3000℃) transition metal compound-based composite ceramic system. In the ultra high temperature (above 2000℃) and oxygen atmosphere of harsh environmental conditions can be used as usual, it has good physical and chemical stability. Mainly used for hypersonic missile, the thermal protection system of space shuttle vehicles, such as wing front, end cap and engine hot end. It is the best alternative of refractory metal, C/C (C/SiC). It is also the most promising materials in ultra high temperature field (Ma Baoxia et al, 2013). This paper developed Cf/ZrC-SiC ultra high temperature ceramic matrix composites for the aerospace industry, and strives to break the technical problem.

2 EXPERIMENTAL MATERIALS AND EXPERIMENTAL PROCEDURES

Carbon fiber reinforced ceramic matrix composite material preparation method are general mud dipping hot pressing method, precursor conversion method, chemical vapor infiltration, and reactive melt infiltration method. Due to the reaction infiltration process with short process cycle, it suited for profiled component molding. And it also reflects the superiority in the low cost preparation of continuous fiber reinforced high temperature resistant ceramic matrix composites. This experiment adopts the reactive infiltration preparation of Cf/ZrC-SiC high temperature resistant ceramic matrix composite method.

2.1 *Experimental material*

The t high temperature resistance Cf/ZrC-SiC ceramic matrix composites materials were prepared by the reaction infiltration. It applied to the ceramic precursor, infiltrating metal and fiber. The main materials are applied to the ceramic precursor, infiltration metal and reinforcing fibers. The ceramic precursor is polycarbosilane (PCS) and phenolic resin.

The infiltration metal is Zi-Si. The zirconium silicon is 10:1. The average diameter of the carbon fiber reinforced is 7um.

2.2 *The experimental process*

The main process of this experiment is divided into two parts. The first part is the preparation of the Cf/C-SiC substrate. The second part is the reaction of the infiltration process. Preparation method of Cf/C-SiC substrate commonly used PIP method. It involves in fiber forming, impregnation of precursor solution, and high temperature cracking. The specific procedure is shown in Figure 1.

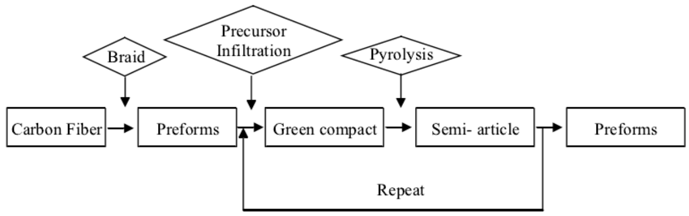

Figure 1 PIP preparation process chart

The substrate placed in a graphite crucible and alloy ingot placed on Cf/C-SiC substrate before the infiltration. It may ensure alloy excess according to the quality of free carbon in the substrate. The thermal insulated for a period of time, then cooling in the furnace. The diagram was shown in Figure 2.

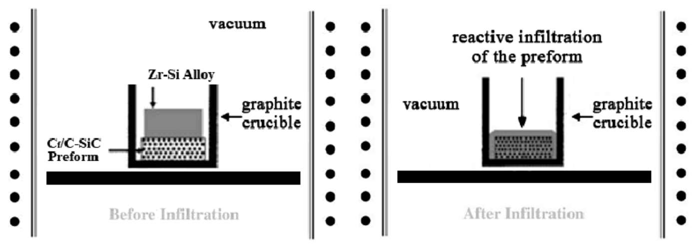

Figure 2 Infiltration process diagram

3 CHARACTERIZATION METHOD

3.1 *Mechanical properties test*

The bending strength and shear strength in universal material machine using three point bending method. In order to reduce the error, each data are measured in a sample of three and took the averaged. The principle is shown in Figure 3.

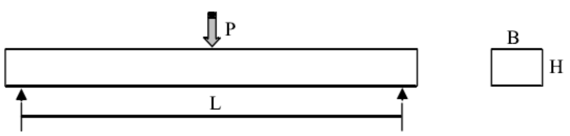

Figure 3 Schematic diagram of flexural strength test

The calculation formula for the flexural strength:

$$\sigma_b = \frac{3PL}{2bh^2} \tag{1}$$

In this formula: σb- three-point bending strength (Mpa);

P- the maximum load of fracture (N);

L- span (mm);

b- specimen width (mm);

h- specimen height (mm).

The elastic modulus of E composites is calculated by the load displacement curve. The calculation formula is as follow:

$$E = \frac{KL^3}{4bh^3} \tag{2}$$

K is the load displacement curve. Other parameters are as the formula (1).

3.2 *Determination of thermal physical properties*

The sample Cf/ZrC-SiC composite along the length direction parallel to the fiber processed into the size of 5mm x5mm x 25mm. At the same time, it should to ensure the parallelism of both ends of the side. In the test, it used the small sample clamped at both ends. In the process of heating, it measured sample temperature coefficient of linear thermal expansion. The sample is cut into the diameter of 12.5mm, thickness of 3 ~ 4mm size of the wafer. It used the laser pulse method to test composite thermal diffusion coefficient.

3.3 *Anti erosion performance test*

It tested the erosion resistance of samples according to the GJB 323A-96 standard. It used the oxyacetylene ablation method. The ablation resistance performance was showed as the material quality of material ablation rate and linear ablation rate. Figure 4 is a schematic diagram of the oxygen acetylene ablation experimental setup.

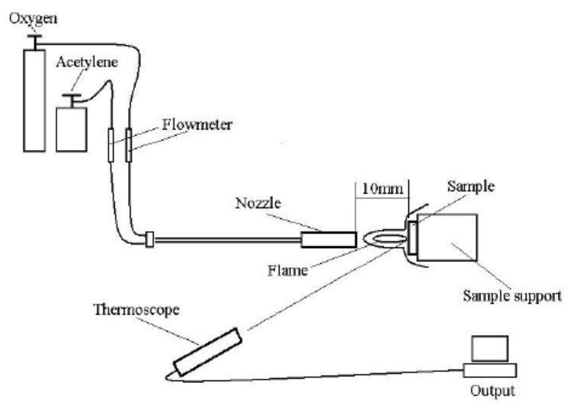

Figure 4 Schematic diagram of oxyacetylene ablation equipment

The mass ablation rate refers to the quality of ablation in unit time sample amount. The calculation formula is as follow:

$$MA = \frac{m_0 - m_1}{t} \quad (3)$$

In this formula: MA- mass ablation rate (g·s-1);

m0- the sample quality before ablation (g);

m1- the quality of specimen after ablation (g);

t- the ablation time of assessment (s).

The linear ablation rate refers to the amount of ablation in the thickness direction at unit time. The calculation formula is as follow:

$$RA = \frac{l_0 - l_1}{t} \quad (4)$$

In this formula: RA- linear ablation rate (mm·s-1);

l0- the thickness of the specimen before ablation (mm);

l1- the thickness of the specimen after ablation (mm);

T- the ablation time (s).

4 RESULTS AND ANALYSIS

4.1 *Study on mechanical properties of Cf/ZrC-SiC Composites*

This experiment studied the influence of infiltration temperature and infiltration time on mechanical properties of Cf/ZrC-SiC Composites. The results are shown in table 1 and table 2.

Table 1 The mechanical properties of different infiltration temperature Cf/ZrC-SiC materials

Temperature /℃	Time/min	Flexural Strength /MPa	Flexural Modulus /GPa
1600	120	100.76	32.78
1700	120	106.83	46.63
1800	120	103.52	32.12

Table 2 The different infiltration time of mechanical properties Cf/ZrC-SiC materials

Time/min	Temperature /℃	Flexural Strength /MPa	Flexural Modulus/GPa
60	1700	103.31	35.43
120	1700	106.83	46.63
240	1700	183.87	54.21
360	1700	201.87	65.34

With the increase of infiltration temperature, the bending strength of composites changed little. It was about 100MPa. The bending modulus at 1700℃ to obtain the maximum value which was 46.63 GPa. The Cf/ZrC-SiC composite three temperatures in the preparation process, a large number of ZrC matrix existed in fiber bundles. Base load transfer capacity decreased. There are pores in the matrix and fiber bundles, it weakened ability of resistance deformation. The bending properties of Cf/ZrC-SiC composites obtained were low (Wang Lingling et al, 2015).

The bending properties of Cf/ZrC-SiC materials with increasing melt infiltration time prolonged. When it extended from 120 min to 240 min, its performance increased. Compared with the infiltration time is 240min, its performance slowdown when the infiltration time is extended to 360min. The infiltration time prolonged, it melt to impregnate fiber bundles and to improve the degree of dense material. It is also beneficial for its mechanical properties.

Comprehensive table 1 Table 2 shows that when the infiltration temperature is 1700℃, the infiltration time is 6h, the flexural strength and flexural modulus of Cf/ZrC-SiC composites were 201.87MPa and 65.34GPa.

4.2 *Research on Thermophysical Properties of Cf/ZrC-SiC Composites*

Cf/ZrC-SiC composite is a new type of high temperature resistant ceramic matrix composites. This new material is mainly aimed at ultra high temperature field. So the research on the thermal properties of materials is the key technology whether this material can be used of some key components.

The thermal expansion coefficient is a key parameter to characterize the thermal stability. It affects the dimensional stability of components in the application. This experiment tested the expansion coefficient of the Cf/ZrC-SiC composite along the generatrix direction from room temperature to 1500℃.Relationship between thermal expansion coefficients of Cf/ZrC-SiC composites with temperature was obtained. As shown in Figure 5.

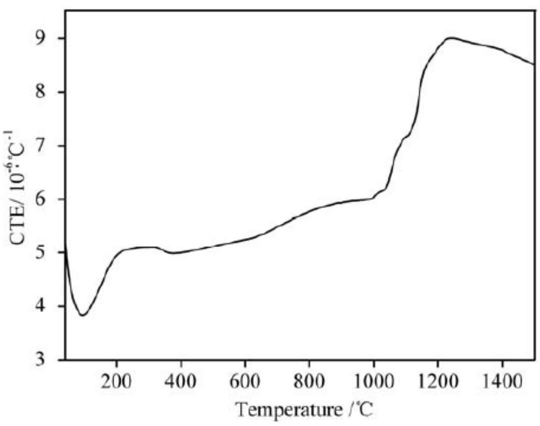

Figure 5 The relationship between Cf/ZrC-SiC composite thermal expansion coefficient and temperature

We can see that with the increase of temperature, the average value of material thermal expansion coefficient along the generatrix direction was increased, coupled with the remainder of alloy materials within the Cf/ZrC-SiC, so the measured thermal expansion coefficient greater.

Thermal diffusivity reflects the heat in the material transfer speed, and the temperature of each part of parametric representation of objects tends to consistent ability. In the same external heating or cooling conditions, the thermal diffusion coefficient is higher. The larger propagation velocity of the internal temperature, the temperature difference is smaller. The Cf/ZrC-SiC composite material at different temperatures the thermal diffusion coefficient is shown in Figure 6. The measured data using two polynomial fittings, it found that thermal diffusion coefficient changes with temperature showed a parabolic law well.

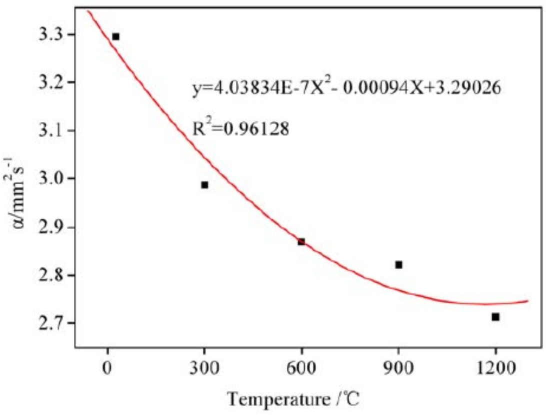

Figure 6 Cf/ZrC-SiC composite thermal diffusion coefficient with the change of temperature

The thermal diffusion coefficient of the composites increased with temperature gradually decreased. In 600℃, it decreased faster. It remained stable at 1200℃. This is a principle that the anisotropic characteristics of Cf/ZrC-SiC composites, and the Cf/ZrC-SiC composite material internal temperature tends to be uniform under high temperature condition. It relieved internal heat stress, and improved the seismic performance of materials (Jin Hua et al, 2013).

4.3 *Study on anti ablation properties of Cf/ZrC-SiC composites*

Preparation of novel Cf/ZrC-SiC resistant ultra high temperature ceramic composite target is expansion of the application of Cf/SiC materials the high temperature. So it studied on the anti erosion performance. According to GJB323-96A standard tests, the ablation resistance test materials used oxyacetylene ablation method. The performance comparison of common Cf/SiC composite with anti ablation Cf/ZrC-SiC samples which infiltration temperature is 1700 and infiltration time is 360min (Duan Liuyang et al, 2015). It was shown in table 3.

Table 3 The performance comparison of anti ablation Cf/ZrC-SiC samples with common Cf/SiC composite

Sample	Ablated time /s	Mass loss Rate/g/s	Recession loss Rate /mm/s
C_f/ZrC-SiC	30	0.013	0.022
C_f/SiC	30	0.043	0.062

Compared with Cf/SiC composite, the ablation resistance of Cf/ZrC-SiC composites increased obviously. The mass ablation rate is reduced 4 times. The linear ablation rate is decreased 3 times. Composite ablation rate change is mainly caused by SiO_2 melt and ZrO_2 particles. SiC oxidized to form SiO_2, it forms the glass melt under the low temperature. SiO_2 glass has good fluidity. The flow in the surface oxide layer formed in the barrier layer to prevent oxidizing atmosphere. High temperature resistance of ceramic ZrC's melting point up to 3540℃ and the melting point of ZrO_2 oxidation products at 2700℃. In the environment of the scour erosion, SiO_2 fluid on the molten state has certain pinning effect. It also reduced the melt viscosity of SiO_2. The linear ablation rate and mass ablation rate compared to Cf/SiC material decreased.

5 CONCLUSION

Study on preparation and properties of Cf/ZrC-SiC ceramic matrix composites, it can conclusion that: The mechanical properties of the samples is the best when the infiltration temperature is 1700℃ and the infiltration time is 360min. Cf/ZrC-SiC ceramic matrix composite's thermal expansion coefficient with the increased of temperature raise. The thermal diffusion coefficient decreased gradually until stable, the temperature field inside the Cf/ZrC-SiC composite material tends to be uniform under the high temperature condition. It relieved internal heat stress, and improved the seismic performance of materials. Compared with the common Cf/SiC composite, the ablation resistance of Cf/ZrC-SiC ceramic matrix composites improved.

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