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ARTICLE

# Effects of $Zr_2Cu$ Packing Modification on Microstructure and Mechanical and Ablation Properties of PIP-Prepared C/C-SiC-ZrC Composites

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**Abstract:** To improve the compactness and properties of C/C-SiC-ZrC composites produced by precursor infiltration and pyrolysis (PIP) method, the low-temperature reactive melt infiltration (RMI) process was used to seal the composites using  $Zr_2Cu$  as the filler. The microstructure, mechanical properties, and ablation properties of the  $Zr_2Cu$  packed composites were analyzed. Results show that during  $Zr_2Cu$  impregnation, the melt efficiently fills the large pores of the composites and is converted to ZrCu due to a partial reaction of zirconium with carbon. This results in an increase in composite density from 1.91 g/cm<sup>3</sup> to 2.24 g/cm<sup>3</sup> and a reduction in open porosity by 27.35%. Additionally, the flexural strength of  $Zr_2Cu$  packed C/C-SiC-ZrC composites is improved from 122.78±8.09 MPa to 135.53±5.40 MPa. After plasma ablation for 20 s, the modified composites demonstrate superior ablative resistance compared to PIP C/C-SiC-ZrC, with mass ablation and linear ablation rates of  $2.77\times10^{-3}$  g/s and  $2.60\times10^{-3}$  mm/s, respectively. The “self-transpiration” effect of the low-melting point copper-containing phase absorbs the heat of the plasma flame, further reducing the ablation temperature and promoting the formation of refined  $ZrO_2$  particles within the  $SiO_2$  melting layer. This provides more stable erosion protection for  $Zr_2Cu$  packed C/C-SiC-ZrC composites.

**Key words:** C/C-SiC-ZrC composites;  $Zr_2Cu$  packing; microstructure; mechanical property; ablation property

## 1 Introduction

Carbon/carbon (C/C) composites have low density, high specific strength, low coefficient of thermal expansion, excellent high-temperature stability, and fine thermal shock resistance. They maintain structural strength above 3000 °C, making them suitable for manufacturing solid rocket motor nozzles, throat liners, missile nose cones, and other high-temperature applications<sup>[1-3]</sup>. Unfortunately, C/C composites are prone to oxidation at temperatures above 400 °C, and the oxidation rate increases rapidly with temperature. This reduces their durability and performance, severely restricting their reliability as structural materials at high temperatures<sup>[4]</sup>. Improving the oxidation and ablation resistance of C/C

composites under harsh conditions has become an urgent research focus. Introducing ultra-high temperature ceramics (UHTCs) has proven effective<sup>[5-7]</sup>. ZrC, a representative UHTC, has a high melting point, remarkable oxidation resistance, and commendable chemical stability. It is worth noting that the resulting oxidized product,  $ZrO_2$ , has an exceptionally high melting point of 2770 °C. The generation of a shielding  $ZrO_2$  film during the oxidation process can withstand external corrosive airflow, thereby enhancing the oxidation and ablation resistance of ZrC modified C/C composites<sup>[4,8-9]</sup>. However,  $ZrO_2$  alone is relatively loose, so SiC and ZrC are often paired to modify C/C composites synergistically.  $SiO_2$  has good mobility to seal cracks by

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filling the porous  $\text{ZrO}_2$  framework, forming a dense continuous  $\text{ZrO}_2$ - $\text{SiO}_2$  protective layer<sup>[10-12]</sup>.

Up to now, various approaches, such as chemical vapor infiltration (CVI)<sup>[13]</sup>, reactive melt infiltration (RMI)<sup>[14-17]</sup>, precursor infiltration, and pyrolysis (PIP)<sup>[18-21]</sup>, have been employed to produce C/C-UHTCs composites. PIP is widely used due to its advantages in low-temperature forming, low fiber damage, good material composition designability, and the ability to produce complex molded parts<sup>[22-25]</sup>. However, the long preparation period due to repeated infiltration-pyrolysis cycles and lack of densification caused by high shrinkage during the precursor pyrolysis process degrades the properties of C/C-UHTCs composites<sup>[2]</sup>. Therefore, shortening the PIP process and improving the density of the pyrolytic matrix are essential in ablation resistance research.

To address these issues, new precursors with high ceramic yields have been synthesized and applied to improve the impregnation efficiency of PIP<sup>[26]</sup>. Combining multiple processes, especially PIP and RMI, has also been proposed in Ref. [27]. Liu et al<sup>[28]</sup> studied the C/C-ZrC-SiC surface with a uniformly distributed ZrC-SiC hybrid ceramic coating using PIP-RMI-PIP. Kong et al<sup>[29]</sup> prepared low-density C/C composites for melt infiltration by PIP, using  $\text{Si}_{0.9}\text{Zr}_{0.1}$  alloy as the melt infiltration metal. They shortened the preparation cycle by using RMI to obtain C/C-ZrC-SiC composites with a density of 2.39 g/cm<sup>3</sup> and a flexural strength of 260 MPa. Zhao et al<sup>[17,30]</sup> prepared C/C-ZrC-SiC-ZrB<sub>2</sub> composites by combining B<sub>4</sub>C-phenolic resin slurry infiltration with RMI of ZrSi<sub>2</sub>, with the melting infiltration temperature reaching 2100 °C. Currently, Zr/Si mixed powder or ZrSi alloys with high melting points are mainly used as infiltrators for preparing C/C-SiC-ZrC composites by RMI, resulting in relatively high preparation temperatures and potential erosion of carbon fibers.

ZrCu alloy has a low melting point (1000–1200 °C), indicating a lower impregnation temperature and less fiber destruction<sup>[31]</sup>. In addition, the “heat sink” and “transpirational cooling” effects of Cu can reduce the ablation temperature and improve the ablation properties of the composites<sup>[32]</sup>. Currently, ZrCu alloy is mainly used for preparing C/C-ZrC-Cu composites<sup>[6,33]</sup>, while ZrCu modified C/C-SiC-ZrC composites fabricated by combining PIP and RMI have seldom been described.

In the current study, with the purpose of reducing the preparation temperature and improving the density of the matrix, C/C-SiC-ZrC composites were firstly fabricated using PIP and then Zr<sub>2</sub>Cu infiltration was performed using RMI since Zr<sub>2</sub>Cu is a typical ZrCu alloy with a melting point of 1025 °C. The effects of introducing Zr<sub>2</sub>Cu on the microstructure, mechanical properties, and ablation properties of the composites were analyzed. The corresponding ablation mechanism was proposed.

## 2 Experiment

Fig.1 shows a schematic diagram of the preparation process for Zr<sub>2</sub>Cu packing modified C/C-SiC-ZrC composites. Ini-

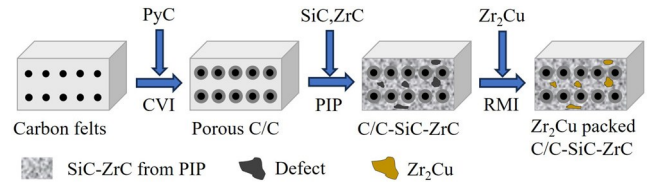


Fig.1 Schematic diagram of preparation procedure of ZrCu-CSiZr composites

tially, 2.5D needled carbon fiber felts, obtained from Yixing Tianniao High Technology Co., Ltd, Jiangsu, China, with a density of 0.45 g/cm<sup>3</sup>, underwent densification via CVI. This process yielded porous C/C preforms with a density of 0.90 g/cm<sup>3</sup>. A mixture comprising polycarbosilane sourced from the National University of Defense Technology, Changsha, China, and zirconium-containing polymer obtained from the Institute of Process Engineering, Chinese Academy of Sciences, Beijing, China, in a mass ratio of 4: 1, was dispersed in xylene to form a homogeneous solution, which served as the SiC-ZrC precursor. Subsequently, C/C-SiC-ZrC composites, designated as CSiZr, were produced through 12 cycles of infiltration-drying-heat treatment. The CSiZr composites were embedded in Zr<sub>2</sub>Cu powder (Eno High-Tech Materials Development Co., Ltd, Qinhuangdao, Hebei, China) within a graphite crucible and then heat-treated under a pressure of 500 Pa. The RMI process was conducted at 1200 °C for 2 h to fully melt and to infiltrate the Zr<sub>2</sub>Cu powder, followed by natural cooling to obtain the Zr<sub>2</sub>Cu packing modified C/C-SiC-ZrC composites, labeled as ZrCu-CSiZr.

The density and open porosity of the samples were measured using the Archimedes method. The mechanical properties were evaluated by a three-point bending test at a loading rate of 0.5 mm/min. The span was 20 mm. A minimum of five samples with dimensions of 30 mm×4 mm×3 mm was tested. The ablation resistance of the samples was evaluated using 35 kW plasma equipment (Metco 9M). The plasma gun tip with 8 mm in internal diameter was extended by 60 mm from the sample. Samples with dimensions of 12 mm×12 mm×12 mm were placed perpendicularly in a flame for 20 s, and the surface temperature during ablation was monitored with an infrared thermometer. Linear and mass ablation rates were determined by the changes in thickness and mass before and after ablation, respectively.

Phase analysis of the composites was conducted by X-ray diffractometer (XRD, AXS D8 ADVANCE). The morphology and elemental composition of samples were examined using scanning electron microscope (SEM, MLA FEG 650) equipped with energy dispersive spectrometer (EDS, Oxford INCA).

## 3 Results and Discussion

### 3.1 Microstructure and composition

The bulk density and open porosity of CSiZr and ZrCu-CSiZr are summarized in Table 1. The bulk density of the composites increases from 1.91 g/cm<sup>3</sup> to 2.24 g/cm<sup>3</sup>, while the

Table 1 Bulk density and open porosity of composites

| Sample     | Bulk density/g·cm <sup>-3</sup> | Open porosity/% |
|------------|---------------------------------|-----------------|
| CSiZr      | 1.91                            | 18.10           |
| ZrCu-CSiZr | 2.24                            | 13.15           |

open porosity decreases from 18.10% to 13.15%. This indicates that PIP C/C-SiC-ZrC composites can be densified quickly and efficiently by Zr<sub>2</sub>Cu packing treatment.

Fig.2 shows the XRD patterns of the composites before and after the packing treatment. Initially, the raw composites contain C, ZrC, and SiC. However, in the XRD pattern of the ZrCu-CSiZr sample, ZrCu peaks are detected instead of Zr<sub>2</sub>Cu, indicating incomplete consumption of zirconium due to its reaction with carbon to form ZrC during the Zr<sub>2</sub>Cu melt impregnation process.

SEM images and EDS results of the composites are displayed in Fig. 3. Fig. 3a presents a representative micro-structure of the CSiZr surface, revealing a considerable

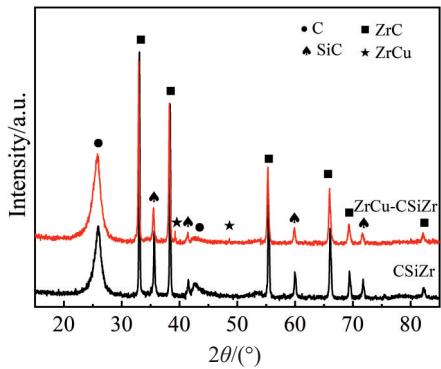


Fig.2 XRD patterns of composites

number of macropores. The cross-section (Fig. 3b) exhibits two main types of pores in CSiZr composites: external macropores (approximately hundreds of microns) between fiber bundles and a few micropores inside the fiber bundles (approximately 10 μm). The presence of these pores is attributed to volume shrinkage caused by gas molecules produced by the organic precursor during the pyrolysis process, making it difficult to achieve a dense ceramic matrix via the PIP process. This restriction is inherent in the preparation of CSiZr composites through PIP<sup>[34]</sup>. After packing, the composites are generally dense without apparent pores (Fig. 3c–3d), indicating that the Zr<sub>2</sub>Cu alloy used for infiltration has appropriate viscosity and good permeability, which can effectively fill the pores, improving the density of the CSiZr composites, as consistent with Table 1. The inset of Fig. 3d shows that the grey compact areas in spot 1 are ZrCu infiltration. To further investigate the matrix and infiltration, area A in Fig. 3c is enlarged, as shown in Fig. 3e, and corresponding element mappings are shown in Fig. 3f–3j. The bright white loose area is the PIP-impregnated SiC-ZrC matrix, while the light grey flat dense area corresponds to melt-infiltrated ZrCu. Notably, the micropores within the PIP SiC-ZrC matrix are unfilled due to the tiny size of these pores, and this issue may be potentially solved by further increasing the melt impregnation temperature.

3.2 Mechanical property

The mechanical properties of the composites are listed in Table 2. Compared with CSiZr, which has a flexural strength of 122.78±8.09 MPa and a flexural modulus of 12.7±3.11 GPa, ZrCu-CSiZr exhibits superior mechanical properties, with a flexural strength of 135.53±5.40 MPa and a flexural modulus of 14.53±1.97 GPa. These improvements are attributed to ZrCu packing.

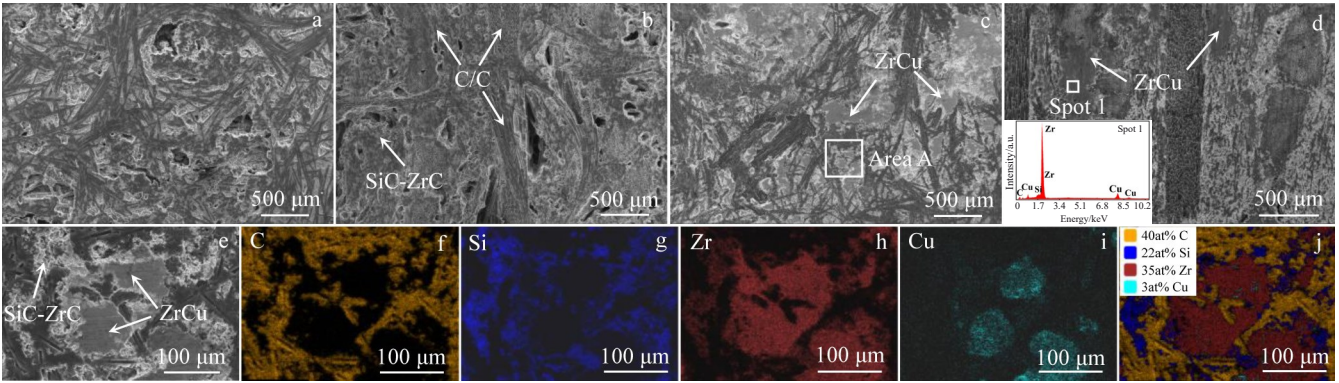


Fig.3 SEM images of surface (a, c) and cross-section (b, d) of composites CSiZr (a–b) and ZrCu-CSiZr (c–d); magnification of area A in Fig.3c (e); EDS element mappings corresponding to Fig.3e (f–j)

Table 2 Mechanical and ablation properties of the composites

| Sample     | Flexural strength/<br>MPa | Flexural modulus/<br>GPa | Linear ablation rate/<br>×10 <sup>-3</sup> mm·s <sup>-1</sup> | Mass ablation rate/<br>×10 <sup>-3</sup> g·s <sup>-1</sup> | Surface<br>temperature/°C |
|------------|---------------------------|--------------------------|---|--|---------------------------|
| CSiZr      | 122.78±8.09               | 12.7±3.11                | 4.32  | 3.09   | 1820                      |
| ZrCu-CSiZr | 135.53±5.40               | 14.53±1.97               | 2.60  | 2.77   | 1750                      |



Load-displacement curves of the composites, illustrating pseudoplastic fracture behavior, are shown in Fig. 4. As observed, ZrCu-CSiZr can withstand higher bending loads than CSiZr. During the initial stages of flexural loading, the loads applied to both composites increase linearly with displacement, exhibiting elastic deformation behavior. As the load increases, the elastic deformation limits of the composites are reached, leading to deviations from the initial straight line and entry into the inelastic deformation stage, accompanied by a decrease in elastic modulus of the composites. With further increase in flexural load, CSiZr exhibits a zig-zagging drop zone, demonstrating the toughening effect of the fibers in the flexure test (i.e., crack deflection, fiber bridging, and fiber pull-out). In contrast, ZrCu-CSiZr shows a more pronounced short sudden break, with damage closely related to the strength of the interface. This is due to the denser short-cut fiber layer post-Zr<sub>2</sub>Cu impregnation, which increases load-bearing capacity and brittleness. In the later stages of the curve, a “stepped” decrease is observed due to toughening effects such as fiber bridging.

Fracture morphologies of the composites are displayed in Fig. 5. According to Fig. 5a and 5c, the fiber bundles in the non-woven layers of both composites are heavily pulled out. The non-woven layer of the carbon felts is composed of parallel fiber bundles, with slight pores between the individual fibers, allowing only a small amount of pyrolytic carbon to deposit on the fiber surface, and resulting in a weak fiber/matrix interface. During loading, cracks tend to spread along this interface, leading to significant bundle pull-out, and effectively absorbing energy. As shown in Fig. 3, ZrCu impregnation is almost absent in the non-woven layer,

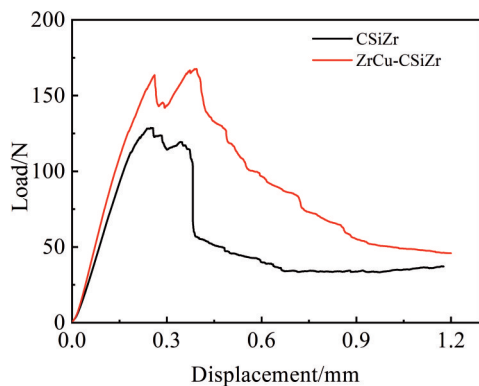


Fig.4 Load-displacement curves of composites

resulting in similar fracture morphologies for the two samples. However, the fracture morphologies of the short-cut fiber webs with ZrCu concentration differ significantly, as shown in Fig. 5b and 5d. CSiZr exhibits a loose distribution of ceramics and winding cracks, indicating relatively low load-bearing capacity. Conversely, ZrCu-CSiZr displays a relatively compact inner matrix with sintered ZrCu, reducing the pore defects and bonding the PIP SiC-ZrC matrix tightly, which can create a strong combination between different phases in the ceramic matrix. Therefore, the composites acquire a more effective ability to resist external loads and to enhance the flexural strength.

### 3.3 Ablation property

The composites were exposed to a plasma flame for ablation tests, as CSiZr composites are typically used to make high-temperature components. The ablative properties of the two composites are shown in Table 2. The linear ablation rate of ZrCu-CSiZr is  $2.60 \times 10^{-3}$  mm/s, which is 39.8% less than that of CSiZr. The mass ablation rates of CSiZr and ZrCu-CSiZr are  $3.09 \times 10^{-3}$  and  $2.77 \times 10^{-3}$  g/s, respectively. Notably, the maximum surface temperature of ZrCu-CSiZr is lower than that of CSiZr, indicating the evaporation of copper during ablation, which contributes to ablative resistance enhancement.

Fig. 6 shows XRD patterns of the composites after ablation, revealing the presence of SiO<sub>2</sub> and ZrO<sub>2</sub>, which are obtained by the oxidation of ZrC and SiC phases. No diffraction peaks containing any Cu phase are detected due to the low copper content and its evaporation during ablation.

SEM images and EDS results, presented in Fig. 7, provide further insights into the ablation behavior of the composites. The surface of ablated CSiZr exhibits a glassy oxide layer with cracks formed during cooling (Fig. 7a). In the magnified view (Fig. 7b), numerous particles are dispersed within the glassy coating. These particles act as pins and enhance the anti-erosion ability of the protective barriers. Analysis of Fig. 7c alongside Fig. 7g–7h indicate that the bright white diffuse particles (1–5 μm) are identified as ZrO<sub>2</sub>, and a dark continuous substance is SiO<sub>2</sub> film. Fig. 7d shows a protective layer of SiO<sub>2</sub>-ZrO<sub>2</sub> based on XRD patterns (Fig. 6) and EDS results (Fig. 7i–7j), which is formed on the ablated surface of ZrCu-CSiZr. The difference is that many small pores result from gas escaping during ablation (Fig. 7e). Notably, small pores in Fig. 7e resulted from gas escape during ablation. Importantly, nano-sized ZrO<sub>2</sub> particles are detected in the SiO<sub>2</sub> protective layer of ZrCu-CSiZr (Fig. 7f), attributed to the cooling effect of ZrCu during ablation<sup>[11,35]</sup>, which enhances

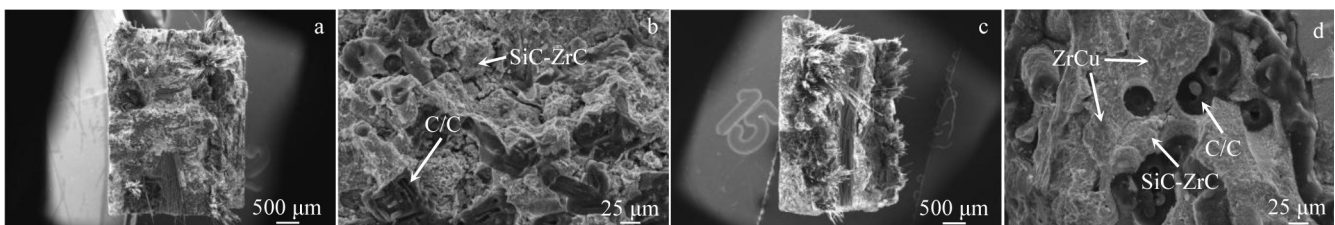


Fig.5 Fracture morphologies of composites: (a–b) CSiZr and (c–d) ZrCu-CSiZr

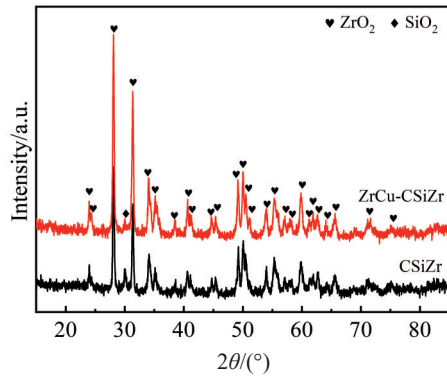
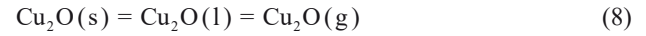
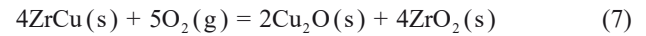
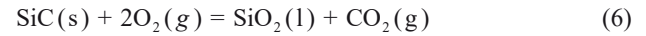
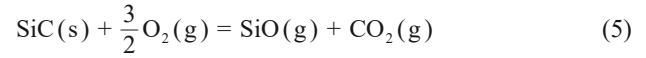
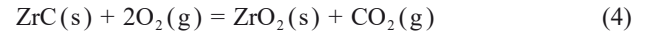
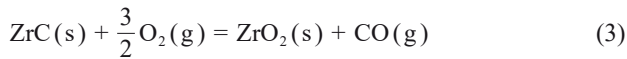


Fig.6 XRD patterns of composites after ablation

pinning effects and reduces  $\text{SiO}_2$  consumption in ablative environment.

### 3.4 Ablation mechanism

When corroded by the plasma torch, the sample surfaces undergo heavy chemical erosion and mechanical denudation (flame scouring)<sup>[23,36]</sup>. The predominant reactions during the ablation process are as follows:



The ablation mechanism of the ZrCu-CSiZr sample is shown in Fig.8. In the initial seconds of ablation, the plasma flame is in contact with the composite, causing a dramatic increase in surface temperature. The various phases in the ZrCu-CSiZr sample begin to oxidize. As previously observed<sup>[13]</sup>, the Gibbs free energy of the oxidation of ZrC and SiC is lower in an oxygen-enriched environment, leading to preferential oxidation to form  $\text{ZrO}_2$  and  $\text{SiO}_2$ . Meanwhile, ZrCu also oxidizes. According to Ref.[37], CuO is converted to  $\text{Cu}_2\text{O}$  at about 1120 °C. The oxidation products of ZrCu are presumed to be  $\text{ZrO}_2$  and  $\text{Cu}_2\text{O}$ , as shown in Eq. (7). As ablation proceeds, the surface temperature of the sample rises, causing  $\text{SiO}_2$  to melt and to form a molten film covering the composite surface. This film resists the erosion of the internal material by external airflow to a certain extent. However, the viscosity of the  $\text{SiO}_2$  melt is low, making it susceptible to being washed away by the ablating flow, which increases the ablation rate. Fortunately,  $\text{ZrO}_2$ , with a higher melting point,

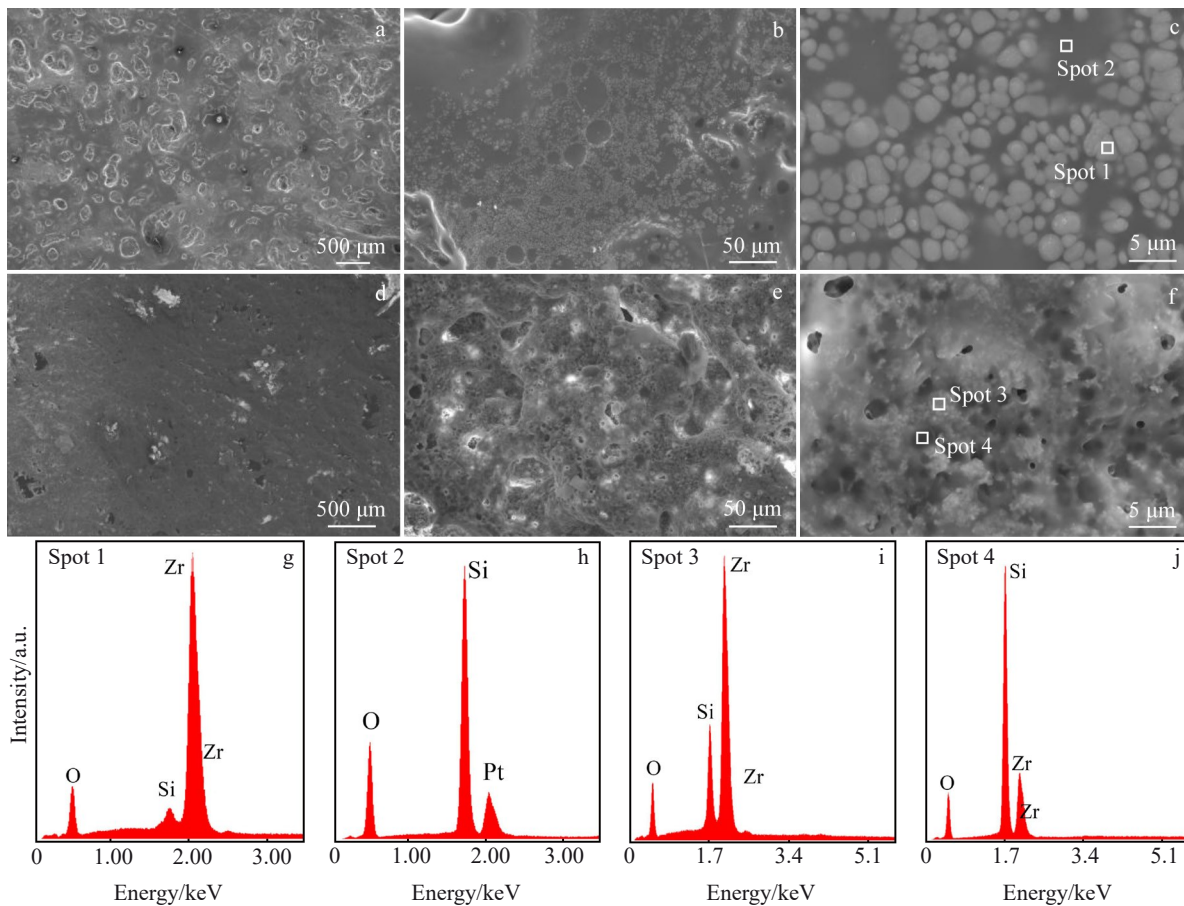


Fig.7 SEM images (a–f) and EDS results (g–j) of composites after ablation: (a–c) CSiZr; (d–f) ZrCu-CSiZr

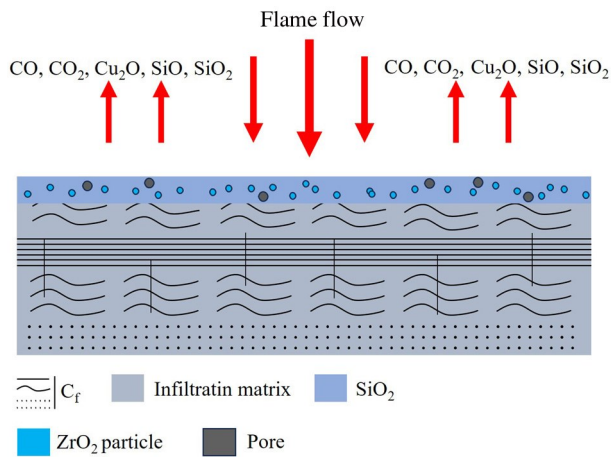


Fig.8 Schematic diagram of ZrCu-CSiZr during ablation

does not melt at the current temperature. It remains as particles embedded in the molten  $\text{SiO}_2$  film, enhancing the stability of  $\text{SiO}_2$  and effectively alleviating the consumption of the molten layer in high-temperature airflow. The gaseous products of these reactions, such as  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{SiO}$ , will escape, causing micropores to form on the  $\text{ZrO}_2$ -fixed molten  $\text{SiO}_2$ .  $\text{Cu}_2\text{O}$  liquefies at about  $1235^\circ\text{C}$  and evaporates when the temperature exceeds its boiling point ( $1800^\circ\text{C}$ ), increasing the number of micropores. Significantly, the heat absorbed by the plasma flame through the evaporation of  $\text{Cu}_2\text{O}$  reduces the surface temperature, inhibiting the growth of  $\text{ZrO}_2$  particles at a lower temperature. This makes the pinning effect more effective, providing adequate protection for the ZrCu-CSiZr composites.

#### 4 Conclusions

1) The  $\text{Zr}_2\text{Cu}$  melt exhibits favorable wettability with the PIP C/C-SiC-ZrC matrix and efficiently fills the macropores, contributing to densification of the composites and a reduction in open porosity of 27.35%.

2) The flexural strength of  $\text{Zr}_2\text{Cu}$  packed C/C-SiC-ZrC composites is improved from  $122.78 \pm 8.09$  MPa to  $135.53 \pm 5.40$  MPa because the denser matrix provides greater resistance to external forces.

3) The ablation properties are enhanced, with mass loss rate and linear recession rate of  $2.77 \times 10^{-3}$  g/s and  $2.60 \times 10^{-3}$  mm/s after plasma ablation for 20 s, respectively. Excellent ablative properties are attributed to stronger pinning of fine nano-sized  $\text{ZrO}_2$  in the  $\text{SiO}_2$  molten protective layer and the volatilization of the Cu-containing phase.

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Zr<sub>2</sub>Cu封填改性对PIP制备的C/C-SiC-ZrC复合材料微观结构和力学及烧蚀性能的影响

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**摘 要:** 为了改善聚合物浸渍裂解法(PIP)制备的C/C-SiC-ZrC复合材料的致密度和性能, 以Zr<sub>2</sub>Cu为填料, 采用低温反应熔体浸渍工艺(RMI)对复合材料进行封孔, 研究了Zr<sub>2</sub>Cu封填后复合材料的微观结构、力学性能和烧蚀性能。结果表明: 在Zr<sub>2</sub>Cu浸渍过程中, 熔体有效地填充了复合材料的大孔隙, 并且由于部分锆与碳发生化学反应, Zr<sub>2</sub>Cu转化为ZrCu。材料的密度从1.91 g/cm<sup>3</sup>增加至2.24 g/cm<sup>3</sup>, 孔隙率降低了27.35%。此外, 封填后的C/C-SiC-ZrC复合材料的抗弯曲强度从122.78±8.09 MPa提高至135.53±5.40 MPa, 等离子体烧蚀20 s后, 与PIP C/C-SiC-ZrC相比, 表现出更优越的耐烧蚀性, 其质量烧蚀率和线性烧蚀率分别为2.77×10<sup>-3</sup> g/s和2.60×10<sup>-3</sup> mm/s。低熔点含铜相的“自蒸发”效应吸收了等离子火焰的热量, 进一步降低了烧蚀温度, 促进了SiO<sub>2</sub>熔化层内细化ZrO<sub>2</sub>颗粒的形成, 这为Zr<sub>2</sub>Cu封填C/C-SiC-ZrC复合材料提供了更稳定的侵蚀保护。  
**关键词:** C/C-SiC-ZrC复合材料; Zr<sub>2</sub>Cu封填; 微观结构; 力学性能; 烧蚀性能

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