

# Frictional response of a novel C/C–ZrB<sub>2</sub>–ZrC–SiC composite under simulated braking

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**Abstract:** A novel braking material, C/C–ZrB<sub>2</sub>–ZrC–SiC carbon fibre-reinforced hybrid ceramic matrix composite, was prepared by chemical vapour infiltration and polymeric precursor infiltration and pyrolysis. Investigation of the microstructure of C/C–ZrB<sub>2</sub>–ZrC–SiC composite showed the homogenous dispersion of three-phase ceramic as the matrix. The frictional properties of the hybrid C/C–ZrB<sub>2</sub>–ZrC–SiC ceramic matrix composite were measured by a disk-on-disk type dynamometer under dry and wet conditions to simulate the normal landing state of aircraft brake disk friction pairs. C/C–ZrB<sub>2</sub>–ZrC–SiC ceramic matrix composite has a higher and more stable friction coefficient under wet condition than under dry condition, indicating that the composite has better performance compared with C/C or C/C–SiC braking materials.

**Keywords:** ceramic matrix composite; brake materials; abrasion; braking testing

## 1 Introduction

Carbon/carbon (C/C) composite was first tested and then replaced conventional metallic or half-metallic brake pads and discs for airplanes in the 1970s due to its excellent tribological properties, low density, high specific heat capacity, and low thermal expansion coefficient among others. C/C composite discs and pads are now widely used not only in airplanes but also in surface vehicles, such as racing cars and rapid trains [1,2]. However, the friction coefficient of C/C brakes has insufficient stability, which is brought about by humidity and oxidation. C/C composite is easily oxidized above 500 °C, which limits its application in vehicles working at elevated temperatures [1–3].

Carbon fibre-reinforced ceramic matrix composite, mainly composed of C/C–SiC composite, has been developed since the 1990s to overcome the disadvantages of C/C composite [4–7]. C/C–SiC composite exhibits superior and more stable tribological properties in comparison with C/C composite, including a lower degree of responsiveness to surroundings and temperatures. The usual method by which SiC is introduced into porous C/C composite is liquid silicon infiltration, in which the liquid silicon reacts with the carbon matrix at high temperatures and forms SiC as the ceramic matrix. However, this technique has the disadvantage of producing residual silicon within the matrix of C/C–SiC composite, which results in unstable friction behaviours during braking [8,9]. Furthermore, the friction coefficient of the C/C–SiC composite can only be adjusted by the volume fraction of carbon and the porosity of the composite. Introduction of other ceramic phases

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ensures broader adjustment of tribological properties of the resulting C/C composite and improves the oxidation resistance, both of which are highly necessary.

In this study, to further improve the braking stability of carbon fibre-reinforced ceramic matrix composite, zirconium diboride and zirconium carbide ceramics are introduced into a matrix. A novel braking material, C/C–ZrB<sub>2</sub>–ZrC–SiC composite, is prepared by chemical vapour infiltration (CVI) combined with polymeric precursor infiltration and pyrolysis (PIP). The frictional behaviours of the composite are determined using a disk-on-disk type laboratory-scale dynamometer under both dry and wet conditions.

## 2 Experiment

### 2.1 Preparation of the composite

C/C–ZrB<sub>2</sub>–ZrC–SiC composite was prepared by CVI with pyrolytic carbon as an interphase in a two-dimensional (2D) carbon fibre preform. The ZrB<sub>2</sub>–ZrC–SiC three-phase ceramic matrix was introduced into the porous C/C composite by PIP technique. Ceramic compositions in the matrix were designed to have a volume fraction of ZrB<sub>2</sub>:ZrC:SiC = 2:1:3. The fabrication steps are as follows. First, 2D needle-punched carbon fibre preform with an initial density of 0.3 g/cm<sup>3</sup>, was obtained. A thin pyrocarbon layer was deposited as an interfacial layer on the surface of these carbon fibres by CVI using 5 kPa methane as the carbon source at 950 °C, from which a porous C/C composite with a density of about 0.65 g/cm<sup>3</sup> was obtained. Thereafter, the hybrid ceramic matrices were introduced into the C/C composite by PIP process. Polycarbosilane (Suzhou Cerafil Co. Ltd., China) was used as the precursor of SiC ceramic. Two other kinds of organic polymers containing Zr and B (synthesized in our laboratory [10]) were used as the precursors of ZrB<sub>2</sub> and ZrC ceramics. Polymeric solutions were prepared by dissolution of the three precursors into dimethylbenzene at a solution concentration of 50 wt%. Then, the C/C composite was dipped into the polymer precursor solution under vacuum (~100 Pa) for 2 h. Pyrolysis was performed in a high-temperature furnace under a flowing argon atmosphere for 2 h at 1500 °C. Further densification was performed by repeated vacuum infiltration of the polymer solution and pyrolysis. The final composite

was prepared after 16 PIP cycles, with a bulk density of 2.06 g/cm<sup>3</sup> and an open porosity of 9.6% (measured by Archimedes' principle with distilled water).

A standard X-ray diffractometer (XRD, D/max-RB) with Cu K $\alpha$  radiation was used to determine the phase-compositions and crystalline state of the prepared C/C–ZrB<sub>2</sub>–ZrC–SiC composite. The morphology of the composite was determined using a scanning electron microscope (SEM, QUANTA 200 FEG) equipped with an energy-dispersive spectrometer (EDS).

### 2.2 Friction test

The braking performance of C/C–ZrB<sub>2</sub>–ZrC–SiC composite was measured on an MM-1000 disk-on-disk type laboratory-scale dynamometer to simulate the normal landing state of aircraft brake disk friction pairs. A pair of brake disks was made from C/C–ZrB<sub>2</sub>–ZrC–SiC composite; the disks included a rotator disk with dimensions of  $\Phi 75$  mm  $\times$   $\Phi 42.5$  mm  $\times$  10 mm (75 mm, outer diameter; 42.5 mm, inner diameter; 10 mm, thickness), and a stationary disk with dimensions of  $\Phi 90$  mm  $\times$   $\Phi 42.5$  mm  $\times$  10 mm. The given angular velocity and contact pressure were 20 m/s and 0.98 MPa, respectively. The test was repeated 15 times under both dry and wet conditions (adding water). The friction coefficient was then calculated. A detailed description of the friction test can be found in the literature [11].

## 3 Results and discussion

The phase compositions of the prepared C/C–ZrB<sub>2</sub>–ZrC–SiC composite are shown in Fig. 1. ZrC, ZrB<sub>2</sub> and SiC ceramic crystals are formed from their precursors after high-temperature pyrolysis. The microstructure of the composite is shown in Fig. 2. The black zones are the carbon fibres covered with a thin pyrocarbon coating and the grey and white zones surrounding the carbon fibres are the complex ceramics. More specifically, the grey zones are SiC particles and the white zones are ZrC/ZrB<sub>2</sub> particles, which can be identified by means of EDS. The complex ceramics in the matrix are integrated together into a homogenous dispersed state. This phenomenon is attributed to the mutual dissolution of the three ceramic precursor solutions used in preparing C/C–ZrB<sub>2</sub>–ZrC–SiC composite at the molecular scale.

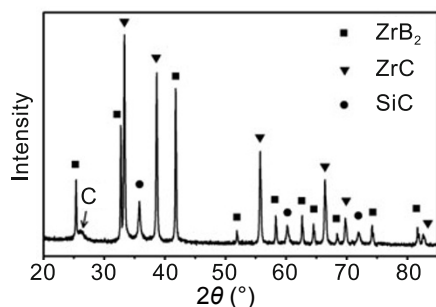


Fig. 1 XRD pattern of the prepared C/C-ZrB<sub>2</sub>-ZrC-SiC composite.

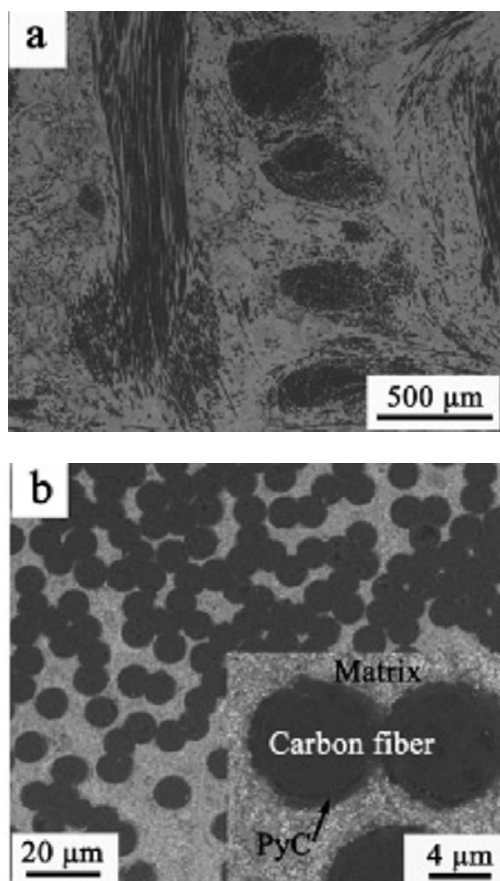


Fig. 2 SEM images showing the morphology of the prepared C/C-ZrB<sub>2</sub>-ZrC-SiC composite: (a) low magnification and (b) high magnification.

The typical braking curves of the prepared C/C-ZrB<sub>2</sub>-ZrC-SiC composite are comparatively shown in Fig. 3; here, the friction coefficient is a function of the braking time under dry and wet conditions. The friction coefficient curve under dry condition reveals a “pre-peak” at the initial stage of braking and a rapidly increasing “tail peak” stage at the end of braking. This “saddle-type” braking curve is consistent with that of previous C/C or C/C-SiC braking materials [11]. The friction coefficient curve

under wet condition also shows a “pre-peak” at the initial stage of braking but no “tail peak” occurring at the end of braking. Moreover, the average friction coefficient obtained during the middle stage under wet condition ( $\mu_{\text{wet}}=0.19$ ) is higher than those obtained under dry condition ( $\mu_{\text{dry}}=0.13$ ), differing from C/C and C/C-SiC composites [12,13]. The worn surface of the discs was also investigated by SEM, as shown in Fig. 4. The carbon fibres coated by pyrocarbon and the hybrid ceramic matrix present soft and hard functions, respectively. Pyrocarbon plays a role of a lubricant to prevent occluding during braking; the hybrid ceramics (ZrB<sub>2</sub>, ZrC and SiC) are used to increase friction resistance; especially, ZrB<sub>2</sub> and ZrC can form rapidly an oxidant layer during braking to prevent pyrocarbon and carbon fibre oxidation. Their combined effects result in more stable braking performance.

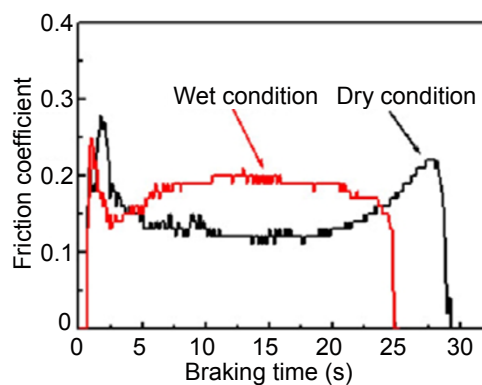


Fig. 3 Typical braking curves of the prepared C/C-ZrB<sub>2</sub>-ZrC-SiC composite with friction coefficients as a function of braking time under dry and wet conditions.

The difference between the braking curves under dry and wet conditions may be due to the solid tribological properties of C/C-ZrB<sub>2</sub>-ZrC-SiC composite. Abrasive grain wear plays a dominant role during the middle stage of braking under dry condition. However under wet condition, water accelerates the oxidation of the composite. The denudation wear of the oxidation film under wet condition has a more significant effect than that under dry condition and results in a higher friction coefficient. At the end of braking, water vaporization under wet condition reduces the surface temperature of the braking discs and weakens the adhesive wear; thus, no “tail peak” occurs until the end of braking. Further analysis and discussion about frictional mechanism of the composite will be carried out in the next research papers.

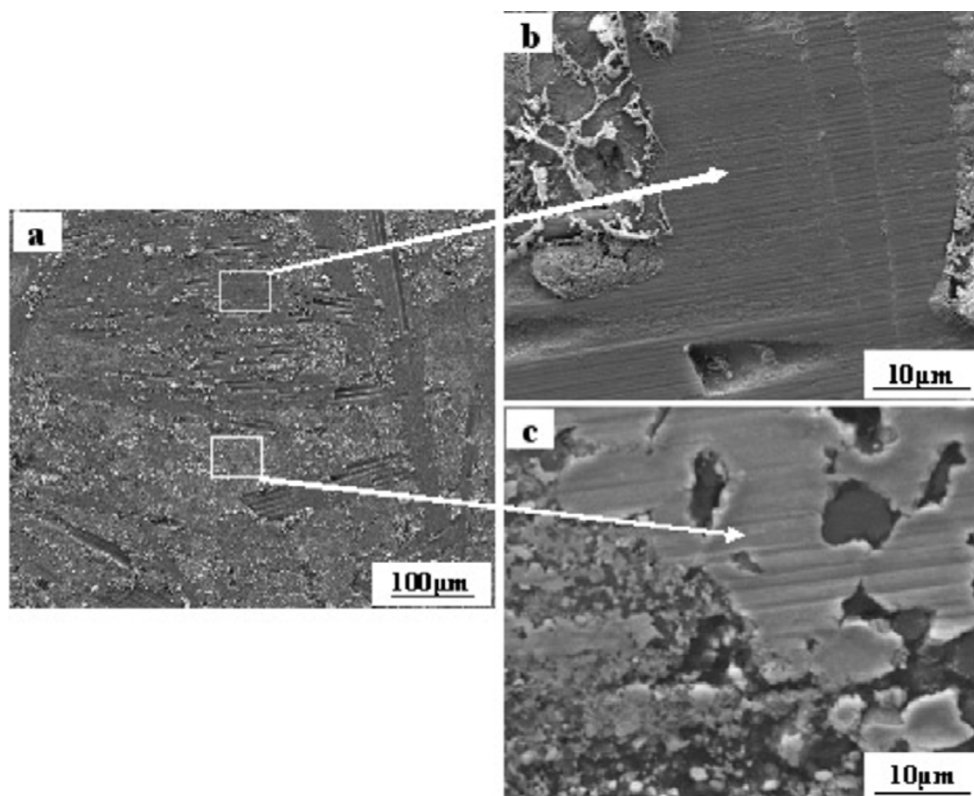


Fig. 4 SEM morphologies of the worn surface under dry condition.

#### 4 Conclusions

A novel braking material, C/C–ZrB<sub>2</sub>–ZrC–SiC composite, featuring a 2D carbon fibre-reinforced hybrid ceramic matrix, was prepared for the first time by CVI and PIP. Microstructure investigation indicated the homogenous dispersion of complex ceramics in the composite. The frictional properties of the composite were measured by a disk-on-disk type laboratory-scale dynamometer under both dry and wet conditions to simulate the normal landing state of aircraft brake disk friction pairs. The braking property of the C/C–ZrB<sub>2</sub>–ZrC–SiC composite under wet condition has a higher and more stable friction coefficient than that under dry condition, indicating that the composite obtained has better performance than C/C or C/C–SiC braking materials. We will test more full frictional and wear properties of this composite and analyse the tribological mechanism in the next work.

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