Microstructure and tribological behaviors of C/C–BN composites fabricated by chemical vapor infiltration

Xiaomeng Fan, Xiaowei Yin*, Yu Cheng, Litong Zhang, Laifei Cheng

Science and Technology on Thermostructural Composite Materials Laboratory, Northwestern Polytechnical University, Xi'an, Shaanxi 710072, PR China

Abstract

In this paper carbon fiber reinforced carbon–boron nitride binary matrix composites (C/C–BN) were prepared by chemical vapor infiltration (CVI). The infiltration of BN in the CVI process was controlled by the diffusion of BCl_3, and BN matrix was distributed homogeneously in the porous carbon fiber reinforced carbon matrix composites (C/C) due to the good infiltration ability of BN. The as-received C/C–BN composites were composed of 92 vol% C and 8 vol% BN. Both the friction coefficient and wear rate of C/C composites decreased significantly by the incorporation of BN. After heat-treated at 1600 °C, the interlayer spacing of CVI BN decreased to 3.36 Å, and CVI BN with high crystalline degree displayed the excellent lubricating effect, leading to the decrease of friction coefficient and wear rate. The improvement of the tribological properties also was partially attributed to the improved oxidation resistance and the formation of friction film by the incorporation of BN matrix.
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1. Introduction

Carbon fiber reinforced carbon matrix composites (C/C) are one of the most promising candidate materials for high-temperature applications because of their intrinsic high thermal stability and low density [1–6]. Based on the research of C/C composites, many works are carried out to modify the properties of C/C composites to further extend their application field [7–11]. The incorporation of SiC in the C/C composites is an effective way to improve their tribological behaviors, and carbon fiber reinforced C–SiC binary matrix composites (C/C–SiC) fabricated by liquid silicon infiltration (LSI) have been widely researched and successfully applied as braking materials at high-speed braking field [12–18]. However, C/C composites with low friction coefficient and low wear rate are required for the potential application in the field of sealing materials and sliding element at high temperatures [1,19]. In order to

decrease the friction coefficient and wear rate effectively, the lubricating additive is introduced into the C/C composites in the present work.

Hexagonal boron nitride (h-BN) has the similar laminate structure like graphite, and also has many similar properties and uses [20]. It has a low hardness (Mohs hardness 1∼2), high thermostability, and better oxidation resistance than graphite [21]. Owing to the weak Van Der Walls force between each layer, h-BN is easily sheared along the basal plane of hexagonal crystalline structure, leading to the excellent lubricating performance of BN [22]. The friction coefficient for Si_3N_4 containing 30 vol% BN against stainless steel reached 0.03 [23], and the friction coefficient of sliding bearings lubricated by the mixture of BN and oil reached as low as 0.015 [24], which all displayed the outstanding lubricating performance of BN.

BN has been introduced into C/C composites by polymer infiltration and pyrolysis (PIP), and carbon fiber reinforced C–BN binary matrix composites (C/C-BN) fabricated by PIP displayed better wear resistance than C/C composites [25,26]. Besides PIP, chemical vapor infiltration (CVI) is another promising process to fabricate

*Corresponding author. Tel.: +86 29 88494947; fax: +86 29 88494620.
E-mail addresses: yinxw@nwpu.edu.cn,
fanxiaomeng@163.com (X. Yin).

ceramic matrix composites. The CVI method has various advantages [27,28]: (1) the process can be conducted at low pressure and low temperature, no external pressure is required, so fiber reinforced ceramic matrix composites with high performance can be fabricated owing to the small residual stress and less damage on the fiber; (2) the phase composition of the matrix can be easily designed by adjusting the kind, concentration, and deposition sequence of the reaction gases; (3) the components with complex shape and high volume content of fibers can be fabricated by a near-net shape process.

However, no literature about C/C–BN composites fabricated by CVI can be found up to now. Therefore, BN was introduced into the C/C composites by CVI to obtain BN modified C/C composites with low friction coefficient and low wear rate in the present work.

In our previous work [29], the microstructure and deposition kinetic of CVI BN were studied, as well as the crystallization degrees of BN annealed at different temperature. In this paper, C/C–BN composites were prepared by CVI, and then the microstructures and tribological behaviors of C/C–BN composites were investigated in comparison with those of C/C and C/C–SiC composites.

2. Experimental

2.1. Fabrication procedure of samples

In this paper, the 3D needled integrated felts with a density of 0.55 g/cm^3 and a fiber volume fraction of 32% were used as the preform. All the carbon types were PAN-based carbon fiber (T300, 12 K, Toray, Japan). The needled integrated felts were fabricated by repeatedly overlapping the layer of 0° non-woven fiber cloth, short-cut fiber cloth and 90° non-woven fiber cloth with needled-punching step by step. The 3D needled carbon fiber preforms were infiltrated by low pressure chemical vapor infiltration (CVI) in a mixture of C$_3$H$_6$ and Ar atmosphere to form C/C composites.

For C/C–BN composites, BN matrix was introduced into the porous C/C composites by CVI for 40 h in a vertical hot-wall reaction furnace with a total pressure of 1000 Pa at 1000 °C. The fluxes of inlet gas were selected as follows: BCl$_3$ = 20 ml/min, NH$_3$ = 60 ml/min, Ar = 50 ml/min and H$_2$ = 100 ml/min. The as-received C/C–BN composites were heat-treated at 1600 °C in nitrogen atmosphere for 2 h. After heat-treated at 1600 °C, the interlayer spacing of CVI BN decreased to 3.36 Å [29], which was lower than that of CVI BN heat-treated at lower temperature. The smaller interlayer spacing of BN means the stronger atomic bonding, leading to the higher moisture resistance. When the CVI BN heat-treated at 1600 °C was exposed to 50 vol% H$_2$O/ 50 vol% Ar atmosphere at 700 °C for 10 h, the sample had no weight variation owing to the good moisture resistance of CVI BN heat-treated at 1600 °C.
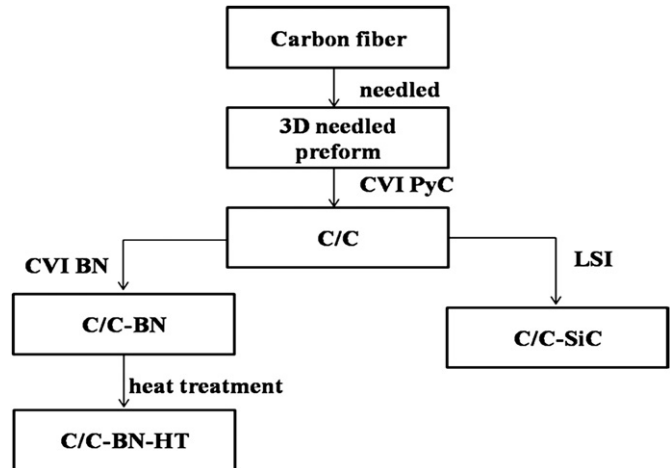

Fig. 1. The schematic of the fabrication procedures of composites.

For C/C–SiC composites, the porous C/C composites were infiltrated with liquid silicon directly in a vacuum furnace at 1500 °C for 0.5 h, and a dense material was obtained. The manufacturing process of composites is presented in Fig. 1. The infiltration of silicon in the carbon material during LSI process has been discussed in the previous literatures [30,31], and the liquid silicon infiltrated the porous composites spontaneously by the capillary force [32], so no detailed discussion on the infiltration ability of liquid silicon in the porous C/C composites appears in this paper.

2.2. Friction and wear test

Friction and wear properties of all the samples were performed on a disk-on-disk MM-1000 testing machine. The tests were carried out with a rotating disk of Φ76 mm × Φ52 mm × 12 mm (76 mm in outer diameter, 52 mm in inner diameter and 12 mm in thickness) and a stationary disk of Φ90 mm × Φ55 mm × 12 mm. Braking test was performed under a moment of inertia of 0.235 kg m^2 with a braking speed of 28 m/s and a braking pressure of 0.8 MPa. Each braking test was repeated for 20 times under the same braking condition. The weight wear rate was calculated by measuring the weight of disks before and after braking tests divided by total number of braking tests. The friction coefficient is calculated using Eq. (1).

$$M = \mu(r_1 + r_2)P/2 \tag{1}$$

where M is the moment, μ is the friction coefficient, P is the braking pressure, r_1 is the inner radius and r_2 is the outer radius.

2.3. Characterizations

Phase analysis was conducted by X-Ray Diffraction (XRD), via a computer-controlled diffractometer (X'Pert Pro, Philips, Netherlands) with CuKα radiation at 40 kV and 100 mA. Data was digitally recorded in a continuous