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Friction and wear properties of pitch/resin densified carbon-carbon composites used for airbrakes①

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[Abstract] By use of X-ray diffractometry and scanning electron microscope (SEM), the friction and wear results obtained from MM-1000 dynamometer tests of CVI pitch/resin C/C composites were analyzed. By investigating the factors that affected the friction and wear properties, such as matrix carbon, application environment, graphitization degree and brake pressure, etc, friction and wear mechanism of carbon materials were probed. The results indicate that pitch densified CVI initially treated composite is more graphitizable with its graphitization degree up to 62%, and which results in uniform small debris easier to generate, more smooth friction curves with the coefficient of 0.3~ 0.4 and relatively higher linear wear and mass loss, compared with CVI/resin C/C composites. It was further proved by SEM observation that tribological behavior of C/C composite was system dependent. Factors determining the friction and wear properties such as the size of debris and its influence on friction and wear, brake pressure, graphization degree and debris film formation interacted and affected each other. The friction and wear mechanism of C/C composites under different high temperature treatments needs further research.

[Key words] C/C composites; friction; wear debris; pitch; resin
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1 INTRODUCTION

Thanks to their low densities, outstanding thermal and mechanical properties and unique self-lubricating capability as well, carbon-carbon(C/C) composites have been widely used as brake materials in aircraft and sports-motor industries besides applications in aeronautics, aerospace or military industry[1]. In fact, C/C composites have been used as friction materials in aircraft brakes for over 20 years, which represents one of the most significant and mature markets for carbon-carbon composites[2]. Many military aircraft, such as the U S F-series and French Mirage fighters and commercial aircraft, such as Boeing series, Airbus, the Concorde, the Canadair Challenger, and Gulfstream III, have installed carbon-carbon (C/C) composites brakes[3,4]. However, long-term fabrication by CVI(chemical vapour infiltration) results in C/C composites' high cost, even if the advantages of the sintered metal brakes replaced by C/C brakes are obvious. Correspondingly, after CVI to a certain density, C/C composites can be quickly densified by impregnation of organic binders, such as pitch and resin, which maybe an effective and cost-spare fabrication process in the future. Some properties about impregnants and aforementioned fabrication process have been investigated in other papers[5~7]. Although C/C composites have been used as aircraft brakes for years, few results have been published in the references, especially regarding to the friction and wear performance of these composites. As a matter of fact, the tribological properties of C/C composites are susceptible to a lot of factors, such as C/C composites' inherent microstructure (e.g. fiber, matrix), heat treatment temperature, porosity (amount and distribution) and application conditions per se because tribological behaviors are system dependent and time dependent[8]. So the present paper reports friction and wear behavior of two-dimensional(2D) PAN(polyarylonitrile) —fiber CVI/pitch/resin based composites under certain experimental conditions to study the effects brought about by matrix, heat treatment temperature and test conditions.

2 EXPERIMENTAL

2.1 Materials and laboratory scale dynamometer

The carbon-carbon composite specimens were made from polyacrylonitrile-based carbon fibers in a chemical vapour infiltrated matrix which were further densified with liquid pitch or Furan resin by impregnation/carbonization, and then graphitized at 2100 ℃/2300 ℃. The final two-dimensional (2D) composites had the densities of 1.82~ 1.85 g/cm^3 and contained 49% fiber, 43% matrix, and 8% porosity.

The characteristics of friction and wear were studied by a home-made laboratory scale inertia type

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of dynamometer (MM-1000) to simulate braking process, as shown in Fig. 1. A ring-on-ring specimen configuration was chosen for this study. The nominal outer and inner diameters of the ring-shaped specimen were 75 mm and 53 mm, respectively. Initial thickness was 14~16 mm. Experiments under constant brake energy were carried out at 7500 r/min, corresponding to linear speed of 25 m/s. Brake pressure were 0.6 MPa and 1.0 MPa. The linear wear and the mass loss of the specimen were both measured in order to evaluate the material losses owing to mechanical wear and/or oxidation. The linear wear was determined by measuring the change in the thickness of the ring specimen after 10 test runs. The mass loss (per time) of the specimen was average mass loss of ten test runs, determined by an electronic balance with a sensitivity of 0.1 mg. All tests were conducted in ambient air with a relative humidity content of 50%~65% at 28 ℃.

2.2 Analysis of wear debris and graphitization degree of composites

The wear debris produced in the tests was examined using scanning electron microscope (JEOL JSM-5600LV).

The graphitization degree of the resultant composite treated under different temperatures was determined by using the X-ray diffraction (XRD) analysis (Rigaku, Model D/MAX-3C) using Cu K_α radiation (0.15405 nm). The position and widths of the peaks were calibrated with respect to silicon internal standard. The interlayer spacing ($d_{(002)}$) and crystallite size (L_c) were calculated from the Bragg and Scherrer equations, respectively[9]. The graphitization degree was the average values of peak top (002), gravity and FWHM (the full width at half maximum), which were calculated from Maire and Merings equation[10].

3 RESULTS AND DISCUSSION

3.1 Results of graphitization

The present carbon-carbon composite (CVI and pitch/resin-based matrix) was heat treated at 2100 ℃ and 2300 ℃ alone. The density change and results from XRD are listed in Table 1.

Although all the carbonaceous materials follow the same graphitization process, the ability to graphitization of different matrix is different. A clear progression of structural arrangement has been observed during graphitization, as listed in Table 1. The graphitization degree (g/%) results by XRD tests indicate that the graphitizability of specimen A is obviously better than specimen B at either heat treatment temperature. The corresponding change of interlayer spacing ($d_{(002)}$) and crystallite height (L_c) shows the same trend. As for specimen A, the interlayer spacing $d_{(002)}$ decreases from 3.4119 Å to 3.3869 Å when the heat treatment temperature is raised from 2100 ℃ to 2300 ℃. The corresponding crystalline sizes and graphitization level are significantly improved with the increasing of temperature. The same trend to specimen B as well.

As for these two specimens, the initial CVI carbon matrix and fiber are similar, the only difference is the impregnant. On one hand, it is believed that Furan resin derived isotropic glass carbon is non-graphitizable turbostratic carbon which would not be graphitized until 3000 ℃ or higher. But in carbon-carbon composites, during carbonization the

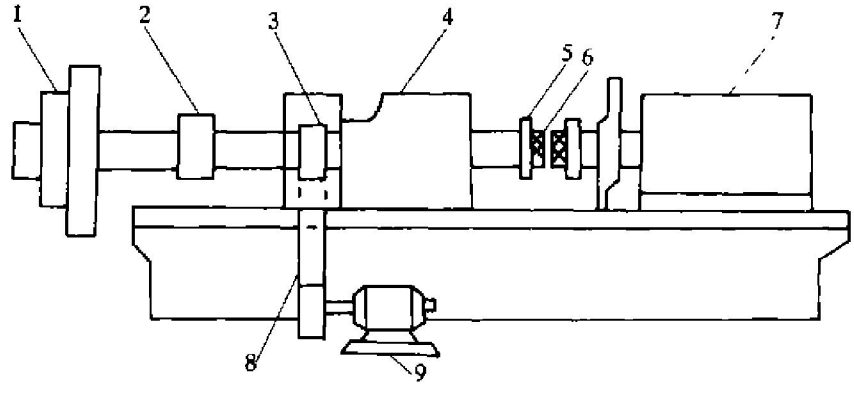

Fig. 1 Schematic diagram of MM-1000 dynamometer

1—Fly wheel; 2—Clutch; 3—Small belt pulley; 4—Rotating platform; 5—Specimen hold; 6—Specimen; 7—Movable pedestal; 8—Belt; 9—Motor

Table 1 Density change and graphitization

Sample	$\rho_{initial}$ (CVI)	ρ_{final}	2100 ℃			2300 ℃		
			$d_{(002)}$/Å	L_c/Å	g/%	$d_{(002)}$/Å	L_c/Å	g/%
A	1.40	1.82	3.4119	85	32.6	3.3869	125	61.7
B	1.41	1.85	3.4222	77	20.7	3.3999	95	46.7

A—CVI pitch-based matrix specimen; B—CVI resin-based matrix specimen; g—Graphitization degree resin shrinks by up to 50% of volume, while the fibers and CVI derived carbon change very little in dimension. It has been postulated that the driving force for the graphization of the "non-graphitizable" matrix is the stress accumulation caused by the difference in the thermal expansion coefficients between the resin-derived matrix, fibers and CVI-derived matrix. Phenomenon is referred to as "stress graphitization"[11]. On the other hand, pitch-derived carbon is more graphitizable in comparison with resin-derived carbon because anisotropic mesophase pitch formed during carbonization process[6], and this would bring about better graphitizability. Probably, the difference of graphitizability between the two specimens might affect their friction and wear properties in some degree.

3.2 Friction and wear properties

Table 2 shows the change of coefficient of friction (μ), linear wear per plane per time, mass loss per time and stability coefficients with the change of brake pressure. Figs. 2, 3 show the typical curves of the friction coefficient as function of brake time for different brake pressures in ambient air.

It is obviously that the friction coefficient of CVI resin-based specimen is a little higher than that of CVI pitch-based specimen under low brake pressure, while the brake curve of the latter is more smooth. The linear wear and mass loss are much smaller under both brake pressures for specimen B.

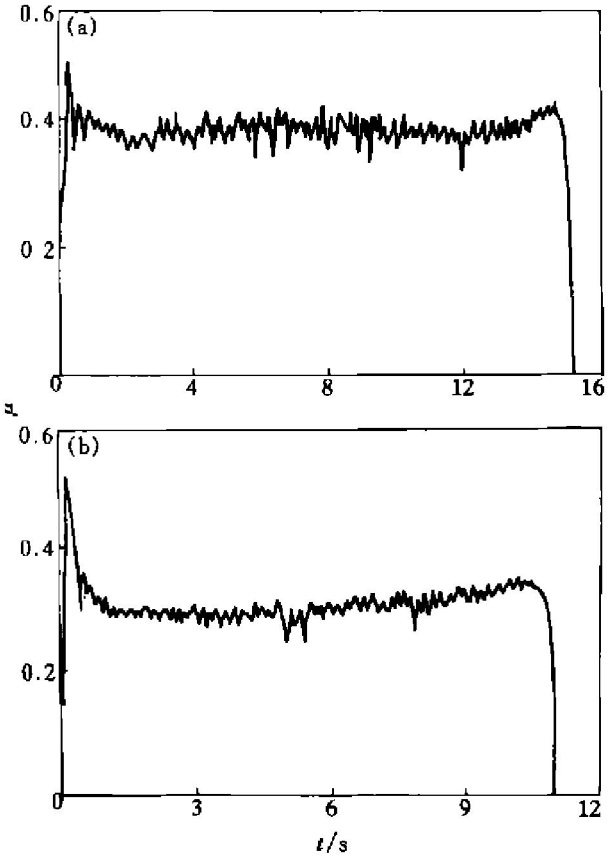

Fig. 2 Typical curves of specimen A(2 300 ℃) showing friction coefficient (μ) dependent on test time (t) under different brake pressures
(a) —0.6 MPa; (b) —1.0 MPa

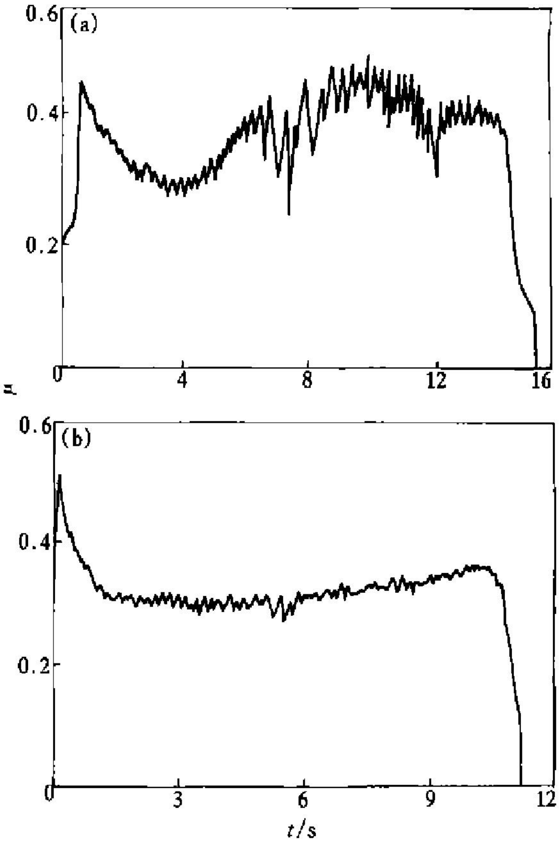

Fig. 3 Typical curves of specimen B(2 300 ℃)
(a) —0.6 MPa; (b) —1.0 MPa

Table 2 Some values of brake simulation tests

Sample	Pressure /MPa	μ	S	Mass loss per number /mg	Linear wear of unit plane of per number/μm
A	0.6	0.37	0.69	12.61	2.28
B		0.39	0.66	3.83	0.65
A	1.0	0.31	0.64	20.06	2.20
B		0.30	0.58	5.42	0.65

S —Stability coefficient

3.3 SEM micrographs of wear debris

Fig. 4 and Fig. 5 are SEM micrographs showing the typical microstructures of the debris in sample A and B (2 300 ℃) at different brake pressures, respectively. The size of the debris generated at high brake pressure for both specimens is smaller definitely.

3.4 Effects of some factors

In spite of limited studies concerning to the friction and wear mechanism of C/C composites, it is now extensively believed that tribological properties of carbon materials are system-dependent[8], i.e., such properties are not determined by carbon materials alone, and some parameters change with ambient conditions.

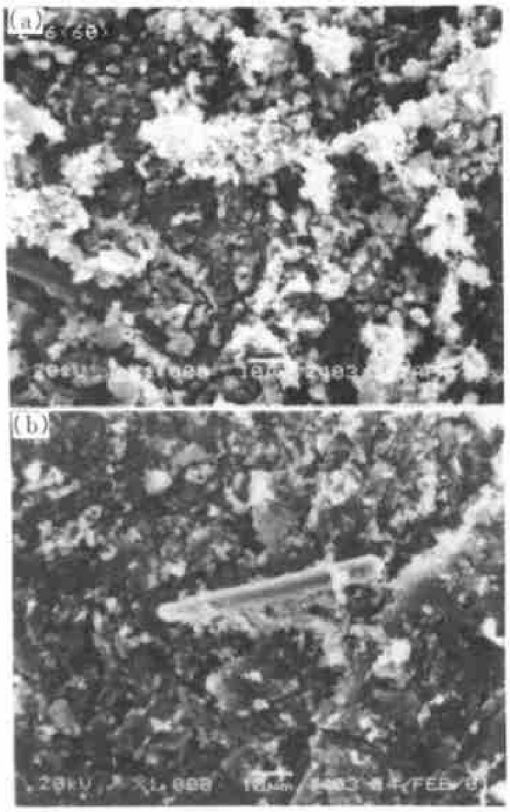

Fig. 4 SEM micrographs of wear debris (specimen A, 2 300 ℃)
(a) —0.6 MPa; (b) —1.0 MPa

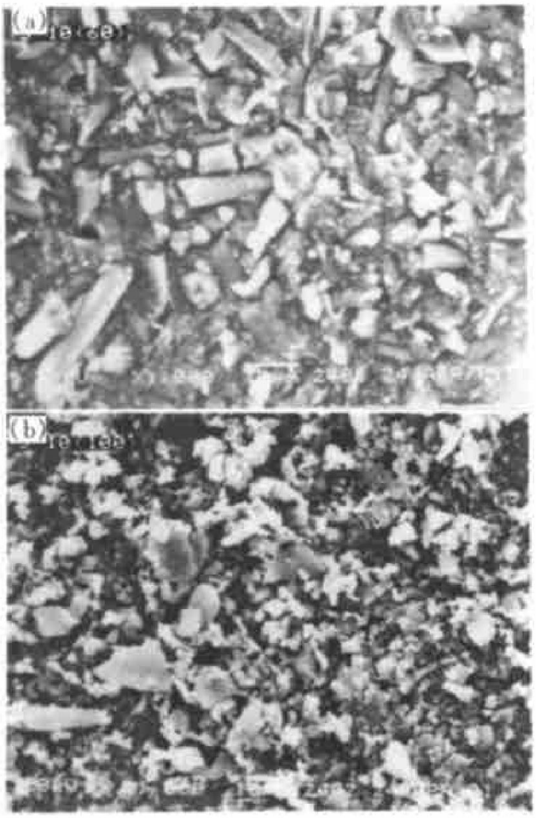

Fig. 5 SEM micrographs of wear debris (specimen B, 2 300 ℃)
(a) —0.6 MPa; (b) —1.0 MPa

Firstly, friction and wear behavior are affected by the environment in which they are performed. In vacuum and inert environments, high friction and wear behavior known as "dusting" wear, the wear rate of carbon materials during dusting is typically 10^2 to 10^4 times higher than that normally encountered in ambient air[12]. It is generally believed that the interaction between the dangling covalent bonds of carbon atoms created by wear is responsible for this high friction and wear behavior. Comparatively, performing in ambient air, due to water absorption at the temperature lower than 185~ 200 ℃, which could act as lubricants and this resulted in low friction and wear. The initial low parts of the curves (Fig. 3(a)) might be attributed to this reason. As the braking process continued, once the temperature of specimens is higher than 185~ 200 ℃, the subsequent desorption of water vapour from the friction surface would undergo a "normal wear" to high dusting wear if there is no debris film formed during that process, and so that a coefficient peak appeared (Fig. 2(a), Fig. 3(a)). Since the chemisorption of oxygen can partially shield the interaction between the dangling covalent bonds of carbon atoms at temperature only up to 650 ℃[13], due to desorption of oxygen another higher peak would occur once the specimen temperature exceeds 650 ℃ if there was not a stable film, which is shown in Fig. 3(a). The fact that this occurred only under light load and low brake pressure would indicate that it is possible due to an insufficient contact pressure to trap the debris between the stator and rotor specimen or might be ascribed to the reason why it is difficult for the specimen to generate debris. As a result, the rubbing interfaces would increase the direct contact between the specimens and which would increase the wear rate and bring about vibration to the brake curves (Figs. 2(a), 3(a)). Hence the brake pressure should be offered to discussion additionally.

Secondly, effects of brake pressure deserve discussion. As a matter of fact, for both specimens A and B, when the brake pressure is increased, friction coefficients decrease but mass loss increases in some degree. Furthermore, the friction curves become more smooth without " initial low value gorge" (Fig. 3(b)). This might be attributed to the generation of loose debris and the subsequent compaction of this debris into a film covering the rubbing surface under higher brake pressure. Perhaps it is the reason why, on one hand, under high initial speed, high brake pressure causes the initial heating rate to be much higher and thus markedly shortens or eliminates the initial low friction regime. On the other hand, high brake pressure bring about larger contact area and stable debris film, which make the brake process more stable.

Thirdly, effects of different matrix, compared with hard resin-derived glassy carbon, pitch derived matrix has thermoelastic properties. It has been shown in Table 1 that pitch-based composites is more graphitizable as a result of higher L_c and smaller $d_{(002)}$. The shear modulus of carbon material is much susceptible to graphitization degree. It increases rapidly with increasing microstructure disorder. Therefore, it is more likely that the response of pitch derived matrix to the shear stresses during braking test is to generate large amount of uniform fragments (Fig. 4(a)), and the particulate debris is partly transformed by shear processes into a friction film. Hence the curve of specimen A under 0.6MPa is more smooth (Fig. 2(a)). With contact pressure increasing, the debris film is comprised of finer worn particle of fiber, pitch, and CVI derived carbons because high pressure could assist deformation of wear particles. Additionally, the higher pressure induces high temperature during braking process and enhances plastic deformation of the wear particles to form the film. This fine debris film acts as a solid self-lubricant which is thought to contribute to the relatively lower friction coefficients and linear wear of specimen A. Repeated delamination of part of the film, formation of fresh wear debris by abrasion of the substrate, and especially oxidation have caused the relatively higher mass loss and linear wear compared with resin-based specimen B. As for resin derived matrix, low graphitization level leads to higher shear modulus, with energy accumulating, parts of the brittle resin matrix fractures abruptly and these large hard particles are not easy to form a smooth film (Fig. 5(a)). The larger the debris, the higher the friction coefficient[14]. In case that it is hard to produce fresh debris, the mass loss and linear wear are lower than that of specimen A, but the curve vibrates more intensely. On the contrast, under higher brake pressure, big particles are ground to finer debris (Fig. 5(b)) and a smooth film forms as a self-lubricant, so that low friction coefficients generates. The same reason as appeared under low pressure new debris film is also hard to regenerate, so the linear wear and mass loss keep low. In a word, CVI/pitch/based composites has better friction properties except for high mass loss under different brake pressure.

As shown in Table 1, different HTT leads to different graphization degree, and this would surely affect the friction and wear of test targets, which would be discussed later.

4 CONCLUSIONS

1) CVI/pitch/based matrix-fibre reinforced composites is more graphitizable than CVI/resin/based composites.

2) Higher brake pressure leads to lower friction coefficients with more smooth friction curves.

3) CVI/pitch derived specimen can generate uniform smaller debris during brake process and which has better friction properties but higher mass loss and linear wear.

4) SEM micrographs of debris indicate that easy to generate uniform debris helps to form debris films which enhance the stability of friction curves.

5) Friction and wear mechanism research indicates that tribological properties of carbon materials are determined by a lot of factors synthetically.

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