Effect of a post-treatment on tribological behavior of PAN/CVI and pitch/phenolic/CVI-based C/C composites

H. H. KUO, J. H. CHERN LIN, C. P. JU*
Department of Materials Science and Engineering, National Cheng-Kung University, Tainan, Taiwan, Republic of China
E-mail: cpju@mail.ncku.edu.tw

Due to their light weight, excellent high-temperature mechanical properties, chemical inertness and self-lubricating capability, carbon/carbon (C/C) composites have been used as aircraft brake material for decades. The C/C composites, most often used as aircraft brake disks, are comprised of either polyacrylonitrile (PAN)-based carbon fiber (fabric laminates)-reinforced chemical vapor infiltrated (CVI) matrix or mesophase pitch-based carbon fiber (chopped yarns)-reinforced phenolic resin char-CVI hybrid matrix composites [1]. Much research has been devoted to investigation of the relationship between tribological behavior and testing parameters, such as sliding speed, break-in speed, humidity, load, surface condition and additives, of a series of two-dimensional (2D) C/C composites [2–9].

The effect of a post-treatment comprising re-impregnation of a carbonaceous additive-doped liquid precursor on tribological performance of a fast-carbonized PAN/phenolic-based C/C composite has been discussed in a previous study [10]. According to the study, after the post-treatment, all samples demonstrate decreases in both COF (coefficient of friction) and weight loss. The results indicated that an appropriate post-treatment may dramatically improve the tribological performance of the PAN/phenolic-based C/C composite. The purpose of the present study is to verify whether this simple treatment is also capable of improving the tribological performance, especially reducing wear rate of two other 2D C/C formulae (PAN/CVI and pitch/phenolic/CVI), which are among the most popularly used C/C composites for aircraft brake disk today. Preparation methods and properties of the pitch/phenolic/CVI (designated "A") and PAN/CVI (designated "B") composites have been described in an early study [11].

The post-treatment was conducted by first impregnating the composites with a carbonaceous additive (1 wt%)-doped liquid precursor in vacuum. Based on the earlier results [10], the two most-promising liquid/additive combinations—furan resin/carbon black (designated "FB") and coal tar pitch/mesophase pitch powder (designated "PM") were selected for the study. The impregnation of the carbon black-doped thermosetting furan resin with a relatively low viscosity was carried out at room temperature. The impregnation of mesophase pitch powder-doped coal tar pitch was conducted at 110 °C to allow the pitch to have a viscosity low enough to infiltrate into the open pores of the composites.

The impregnated composites were placed in an oven at 70 °C for 6 hr to remove excess solvent, followed by curing (160 °C for 30 min in air), post-curing (230 °C for 2 hr in air) and carbonization (1200 °C for 30 min in nitrogen) processes. The carbonized samples were subsequently graphitized to 2200 °C for 30 min in a helium-purged furnace (Model 1000-3060-FP20, Thermal Technology Inc., Santa Barbara, California, USA). Please note that the term "graphitized" is solely used to describe the heat treatment and has no implications as to the resulting carbon crystal structure.

Bulk density and open porosity of the composites were measured using a water saturation method according to ASTM C830. As indicated in Table I, all samples increased in density and decreased in porosity after the post-treatment, as expected. The "PM" samples had larger increases in density than "FB" samples due to the higher density of the pitch precursor.

The constant-speed friction and wear tests were conducted in air with a relative humidity of 50–60% using a homemade disk-on-disk sliding wear tester [10]. For the samples having an outer diameter of 25 mm and inner diameter of 10 mm for wear testing, one unit rpm is equivalent to an average linear speed of 0.055 m/min. A pressure of 1.7 MPa and fixed rotor speed of 1000 rpm, which is equivalent to an average linear speed of 55 m/min, were used for all tests. Such a linear speed is in the range of actual aircraft "taxi" speed. Prior to testing, all samples were mechanical polished to #1200 grit level, followed by ultrasonic cleaning to remove debris on the sample surface. The constant-speed slide testing was performed by first accelerating the rotor sample to the desired speed (1000 rpm). When the rotor speed was reached, the stator sample (same composite under same surface condition) was loaded with a normal pressure of 1.7 MPa against the rotor for 2 min, after which the power to the motor was automatically switched off. Prior to each subsequent test, the samples were allowed to cool to room temperature and their weights were measured. The tests were conducted six times for each sample. The COF, μ, was determined using the equation, $\mu = M/rF_n$, where M

*Author to whom all correspondence should be addressed.

0022–2461

TABLE I Density and porosity data of samples used for the study

Sample code	C/C formula	Bulk density (g/cm^3)	Open porosity (vol%)
A	Pitch/phenolic/CVI	1.73	7.94
A-FB	A-furan/carbon black	1.74	6.20
A-PM	A-coal tar pitch/mesophase pitch	1.80	6.86
B	PAN/CVI	1.73	12.67
B-FB	B-furan/carbon black	1.77	11.16
B-PM	B-coal tar pitch/mesophase pitch	1.78	8.06

is the torque, F_n the normal force and r the average radius of the sample.

As can be seen from Fig. 1, the COF of sample A underwent an abrupt transition during most sliding tests. According to earlier studies [1, 14, 15], during the transition, the initially formed thin, smooth lubricative film was suddenly disrupted and turned into a thick powdery debris layer that caused friction coefficient to abruptly rise. The samples after post-treatment exhibited a much more stable COF. The pre-transitional low COF regime was largely eliminated. Transition only occurred in sample A-FB during the first test, while no transition was observed in any test of sample A-PM. A similar post-treatment-induced friction-stabilizing effect was also observed in B-group samples.

To avoid the effect of pre-transitional low COF values (0.1–0.2) on average COF, Fig. 2 compares the average COF values of last three tests among the six different samples. The post-treatment "FB" did not significantly change (according to one-way ANOVA at the 0.05 level) the average COF of sample A (0.45–0.47), but largely increased the average COF of sample B (from 0.51 to 0.72). On the other hand, the post-treatment "PM" mildly increased the average COF values of samples A and B to 0.58 and 0.56, respectively.

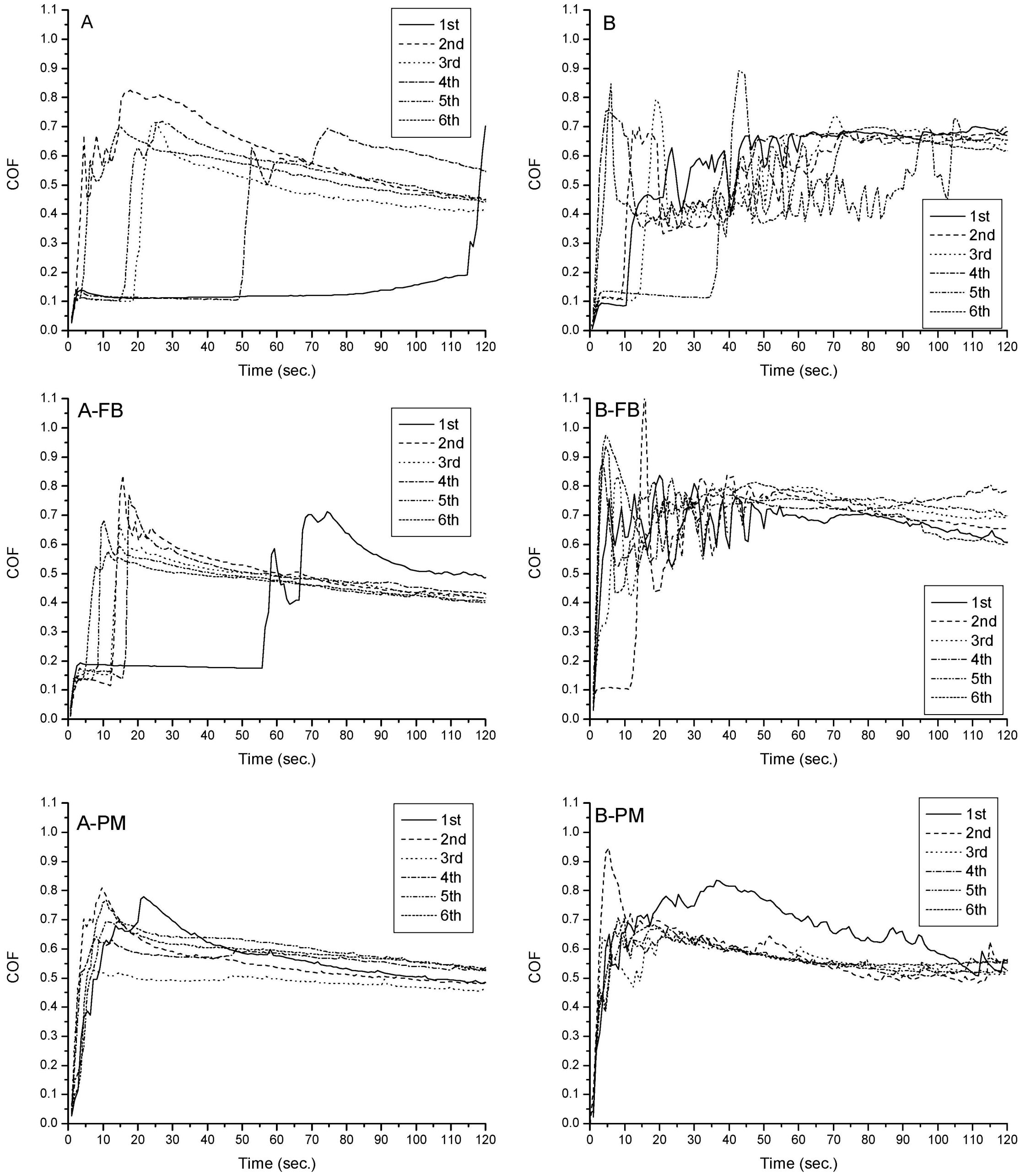

Figure 1 Variation in COF with sliding time of each test.

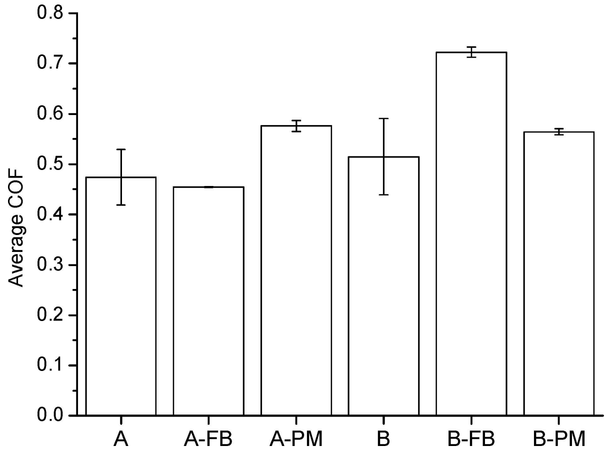

Figure 2 Average COF values of last three tests.

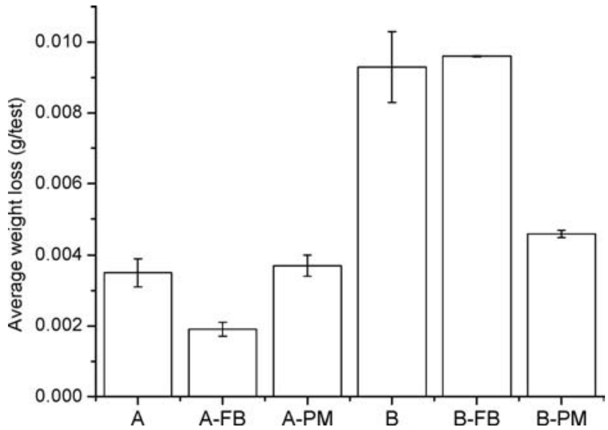

Figure 3 Average weight loss of last three tests.

One most interesting result from the present post-treatment is the dramatic reduction in wear rate of the composites. As demonstrated in Fig. 3, after "FB" treatment, the average weight loss (g/test, of last three tests) of sample A largely decreased from 0.0035 to 0.0019 (by 45.7%). After "PM" treatment, the weight loss of sample B also largely decreased from 0.0093 to 0.0046 (by 50.5%). On the other hand, the post-treatment "PM" did not significantly change the weight loss of sample A, and post-treatment "FB" did not significantly change the weight loss of sample B, either. Another interesting result is that the post-treatment also reduced the variation in average weight loss, as shown in Figs. 2 and 3, which further supports the stabilization effect of the post-treatment on COF.

The result that the weight loss of sample B was much larger than sample A is similar to that of an earlier report [1] and might be explained primarily by the large difference in porosity level between these two composites. However, it seems that the large decreases in weight loss observed in A-FB and B-PM but not in A-PM and B-FB cannot be explained merely by their differences in porosity level. Furthermore, the earlier-mentioned post-treatment-induced friction-stabilizing effect cannot be explained by the changes in weight loss. For example, compared to sample A, the COF of sample A-PM was largely stabilized (Fig. 1) yet the weight loss of the two samples are similar (Fig. 3). It seems that the change in porosity is one of the several factors which could be attributed to the changes in wear rates. Due to the nature of the different post-treatments, it is likely that there are differences in the wear debris formation and tribological behavior, both of which could influence the resulting wear rates.

The present study has demonstrated that post-treatments can stabilize the COF and reduce the wear rates of PAN/CVI and pitch/phenolic/CVI-based C/C composites. The mechanism which causes these effects is clear, but it may result from differences in and process of wear debris generation and the tribological behavior of the wear surface. Nevertheless, from a practical point of view, the present study has suggested a fairly simple approach to not only largely stabilize COF, but also effectively cut down by nearly half the wear rates without changing much the COF values of two major C/C formulae used as aircraft brake disk today.

Acknowledgments

Authors are grateful to the National Science Council of the Republic of China for support of this research under Contract No. NSC 90-2216-E-006-037. Authors also acknowledge the use of graphitization furnace at the Department of Materials Engineering of Tatung University.

References

1. J. D. CHEN and C. P. JU, *Carbon* **33** (1995) 57.
2. *Idem.*, *Mater. Chem. Phys.* **39** (1995) 174.
3. C. P. JU, J. D. CHEN and J. H. CHERN LIN, *J. Mater. Sci. Lett.* **14** (1995) 740.
4. C. P. JU, K. J. LEE and J. H. CHERN LIN, *Wear* **199** (1996) 228.
5. J. D. CHEN, J. H. CHERN LIN and C. P. JU, *ibid.* **193** (1996) 38.
6. *Idem.*, *J. Mater. Sci.* **31** (1996) 1221.
7. K. J. LEE, H. H. KUO, J. H. CHERN LIN and C. P. JU, *Mater. Chem. Phys.* **57** (1999) 244.
8. B. H. MAYER, PhD Thesis, University of Karlsruhe, Germany, 1991.
9. D. S. LIM, J. W. AN and H. J. LEE, *Wear* **252** (2002) 512.
10. H. H. KUO, J. H. CHERN LIN and C. P. JU, *ibid.* (in press).
11. S. E. HSU and C. I. CHEN, in "Superalloys, Supercomposites and Superceramics" (Academic Press, San Diego, CA, 1989) p. 721.
12. C. P. JU, J. H. CHERN LIN, K. J. LEE and H. H. KUO, *Mater. Chem. Phys.* **64** (2000) 196.
13. E. M. TATARZYCKI and R. T. WEBB, in "Friction and Wear of Aircraft Brakes. Friction, Lubrication, and Wear Technology," ASM Handbook (ASM International, 1992) Vol. 18, p. 582.
14. C. P. JU, K. J. LEE, H. D. WU and C. I. CHEN, *Carbon* **32** (1994) 971.
15. J. D. CHEN and C. P. JU, *Wear* **174** (1994) 129.

Received 26 August
and accepted 30 November 2004