EFFECT OF GRAPHITIZATION SEQUENCE ON MECHANICAL AND TRIBOLOGICAL BEHAVIOR OF FAST-CARBONIZED PAN-PHENOLIC RESIN-BASED CARBON-CARBON COMPOSITE

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Introduction

Due to low density, excellent thermal, mechanical, and tribological properties, carbon/carbon (C/C) composites have become the top choice as aircraft brake disk materials. Many advanced fighters, commercial aircrafts, racing cars, and some high energy transports have employed C/C brake disks [1, 2].

An earlier study [3] indicated that the bending properties of a PAN/phenolic-based C/C composite processed at a conventionally low carbonization rate of 1°C/min and at an ultrahigh carbonization rate of 1000°C/min were comparable. Furthermore, the composite carbonized at 1000°C/min had much higher fracture energy than that carbonized at 1°C/min. The sharp increase in carbonization rate can be very economically beneficial to the C/C industry.

Another earlier study [4] indicated that the graphitization temperature of 1900°C would be the most optimal temperature among the three temperatures (1700, 1900, 2100°C). The conventionally-used higher temperature for graphitization (usually higher than 2000°C) is not necessary and would not perform better under the current wear test conditions.

Reported in the present study is the effect of graphitization sequence on mechanical and tribological properties of the same PAN-phenolic type composite fabricated at the same, high carbonization rate of 1000°C/min.

Experimental

The reinforcing fiber and matrix phenolic resin used for the preparation of the present PAN/phenolic-based C/C composite are a randomly oriented chopped (4.5 mm) PAN-based carbon fiber (Torayca T700SC, 12K, Toray Co., Japan) and a powdery phenolic resin (RM-18389, Texxco, Taiwan), respectively. A resole-type phenolic resin (PF-650, Chang Chun Petrochemical Industry, Taiwan) was used as impregnating resin.

The chopped carbon fibers (55 vol%) were first mixed with the matrix phenolic powder (45 vol%) in a mode to form a 110 mm \times 110 mm composite, followed by hot pressing, curing, and post-curing.

Carbonization was conducted by heating the post-cured composite under a nitrogen atmosphere to 1100°C at a heating rate of approximately 1000°C/min. The nitrogen gas was introduced continuously into the furnace at a constant flow rate of 0.6 L/min. The carbonized composite was subsequently graphitized to 1900°C in an argon-purged graphitization furnace. The porous composite was then densified by re-impregnation with the impregnating resin, followed by curing, post-curing and carbonization. To improve density and properties of the composite, four such re-impregnation/carbonization cycles were applied.

To study the effect of graphitization sequence on the properties of the composite, two series of graphitization treatment were used for comparison: (i) Graphitization was conducted following first carbonization treatment (designated "GD" series); (ii) Graphitization was conducted after two re-impregnation/carbonization cycles (designated "DG" series). Furthermore, to learn how the number of densification/carbonization cycle on properties of the composite, to one group of samples were added three additional re-impregnation/ carbonization cycles (GD4 vs. GD7; DG4 vs. DG7).

The flexural strength and modulus of the composite were measured by three-point bending test based on ASTM D790 method with the span-to-depth ratio of 16. Samples for this test were in a size of 50 mm \times 10 mm \times 2.2 mm. A Shimadzu AGS-500D universal tester (Shimadzu Corp., Kyoto, Japan) was operated at a crosshead speed of 1 mm/min with a support span of 40 mm. All the bending test data shown in this paper are the average of five samples.

A simulated-stop wear test was conducted on the composite with a normal pressure of 1.0 MPa and 1200 rpm (equivalent to an average linear speed of 1.1 m/s) in an environment with a humidity level of 45-65%. A homemade disk-on-disk sliding wear tester was used for the test. Prior to testing, all samples were mechanical polished to #1200 grit level, followed by ultrasonic cleaning to remove debris on surface. Prior to each subsequent test, the samples were allowed to cool to room temperature. The coefficient of friction (COF), μ , was determined using the equation, $\mu = M/rF_n$, where M is the torque, F_n the normal force and r the average radius of the sample. The simulated-stop wear test was repeated 40 times under each condition.

Results and Discussion

The flexural strength and flexural modulus values of composite samples processed from different graphitization sequences are demonstrated in Figs. 1 and 2, respectively. As indicated in Figs. 1 and 2, the GD series (GD4 and GD7) exhibited higher flexural strength and flexural modulus values than DG series (DG4 and DG7). For example, the flexural strength and flexural modulus values of composite samples GD7 (69.5 MPa and 31.2 GPa, respectively) were respectively higher than composite samples DG7 (63.7 MPa and 26.5 GPa, respectively). The highest flexural strength and flexural

modulus values of composite, GD7, were directly reflected in its highest bulk density (1.49 g/cm^3) and lowest porosity level (8.75%).

As indicated in Fig. 3, the average COF values of composites processed from different graphitization sequences are similar. For example, the COF values of GD4 and DG4 are 0.37 and 0.36, respectively, while the COF values of GD7 and DG7 are both 0.38. The composites prepared from 7 cycles are slightly higher than those prepared from 4 cycles.

The weight loss values of composite samples processed from different graphitization sequences are shown in Fig. 4. As indicated in the figure, the average weight loss of DG4 (8.2 mg) is significantly larger than those of the other three composites which demonstrate similar weight losses (4.6-?? mg).

The SEM micrographs of composite worn surfaces are demonstrated in Fig. 5.As shown in Figs. 5a, 5b and 5d, a smooth and quite adherent film was on the worm surface. On the worn surface of DG4, however, much more particulate type debris was observed (Fig. 5c), which was probably attributed to its much larger wear as indicated in Fig. 4.

According to these mechanical and tribological data, it was concluded that, for the current C/C formula, the GD treatment would be a better choice than DG treatment.

References

- 1. Ruppe J.P., Can. Aeronaut. Space J., 26 (1980) 209.
- 2. Stimson I.L. and Fisher R., *Philos. Trans. R. Soc. London*, A294 (1980) 583.
- 3. Kuo, H.H., Chern Lin, J.H., and Ju C.P., *Carbon* **43** (2005) 229-239.
- Ju C.P., Chen Y.M., Lin H.Y., Lee K.J. and Lin J.H. Chern, "Effect of graphitization temperature on mechanical and tribological behavior of PANphenolic resin-based carbon-carbon composite", ICCE – 17.

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Fig. 1. Flexural strength values of composite samples processed from different graphitization sequences.



Fig. 2. Flexural modulus values of composite samples processed from different graphitization sequences.



Fig. 3. COF values of composite samples processed from different graphitization sequences.



Fig. 4. Weight loss values of composite samples processed from different graphitization sequences.



Fig. 5. SEM micrographs of composite worn surfaces: (a) GD4, (b) GD7, (c) DG4, (d) DG7