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Carbonization Rate and Impregnating Methods on the Tribological Behavior of Carbon/Carbon Composites

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Abstract

This study compares two different impregnating methods to investigate the influence of carbonization rate on the tribological properties of carbon/carbon (C/C) composites. The experimental results indicate that the bulk density, hardness, infiltration efficiency and friction coefficient of pressure impregnating specimens are higher than those of vacuum impregnating specimens. In comparison with the vacuum impregnating specimens, the pressure impregnating specimens also show the lower apparent porosity and weight loss. Studies on the tribological properties of the specimens with different carbonization rate show that the specimens with the higher carbonization rate exhibit the lower bulk density and infiltration efficiency, but the higher porosity and friction coefficient. These results are attributed by the defect induced by the thermal stress during the higher carbonization rate process. In addition, the pressure impregnating method produces a more notable effect on the properties of C/C composites than that of the carbonization rate.

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Keywords: Carbon/carbon (C/C) composites; carbonization; impregnating; friction; wear

1. Introduction

Due to their low density, excellent thermal and mechanical properties, chemical resistance and self-lubricating capability, carbon/carbon (C/C) composites have become today's top choice of material for aircraft brake disks [1]. However, the high cost of C/C composites, due to the complex manufacturing production processes, has prohibited C/C composites from being considered as candidate materials in domestic vehicles.

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Several studies have investigated the relationship between the tribological behavior and wear parameters of C/C composites. For instance, Awasthi, Wood and Ju et al. [3-9] studied the influences of composite type, wear parameters (loading, sliding speed, etc.), environment (temperature, humidity, etc.) and the prior history of the composite surface on the tribological behavior of C/C composites. To the best of the authors' knowledge, there exists very limited literature on tribological behavior associated with processing parameters of C/C composites. Therefore, in the past decade many engineers focus on understanding the mechanisms of the tribological behavior of C/C in order to control the key parameter for improving the complicated fabrication process [10, 11]. The impregnating method and the carbonization rate considered, known as the crucial parameters on the tribological behavior of C/C composites, were well studied in this paper.

2. Experimental

2.1 Sample preparation

All raw materials, such as chopped PAN-based carbon fibers, phenolic resin, mesophase pitch and carbon black powder were mixed and press-molded to make round disks 25.4 mm in diameter and of desirable thickness. These as-cured composites were then carbonized in the furnace to 1000 °C under nitrogen atmosphere to make preform (undensified) specimens. Preform specimens were further densified using liquid resole-type phenolic resin as the impregnant. Two different impregnation processes, vacuum and pressure impregnation processes were used individually. The impregnation-carbonization cycle of each individual specimen was repeated up to four times. Carbonization was conducted by heating the impregnated specimens in nitrogen atmosphere to 1000 °C. Three different carbonization rate, 3 °C /min、100 °C /min and 1000 °C /min were used individually. Table 1 lists different processing parameters and specimen designation.

Table 1 Process parameters and specimen designation.

Impregnating Method	Pressure			Vacuum		
Carbonization Rate (°C /min)	3	100	1000	3	100	1000
Specimen Designation	P3	P100	P1000	V3	V100	V1000

2.2 Density and porosity

The density and open porosity of fabricated specimens with different densification parameters were measured by the water immersion method according to the ASTM C-20.

2.3 Hardness test

Hardness values of all specimens were determined at HRR (Rockwell hardness R-scales) levels by the hardness testing machine (Akashi, ATK-600, Japan). A 12.7-mm spherical steel ball was used as the

indenter. At least five positions were tested for each set of samples. The experimental hardness data were taken by averaging these test values.

2.4 Friction and wear tests

Friction and wear tests were conducted using a homemade disc-on-disc sliding wear tester (Fig. 1). Prior to testing, all specimens were mechanically polished through a level of #1200 grit paper, followed by ultrasonic cleaning and drying. Multiple continuous wear tests were conducted under the same wear condition in this study. A fixed load of 0.8 MPa, constant rotor speed of 600 rpm (linear speed is 0.399 m/s) and testing time of 300 sec (sliding distance is 120 m) were used in every wear test. Since the rotor and the stator were made of the same material, the mass losses from both discs were measured and were averaged for several runs.

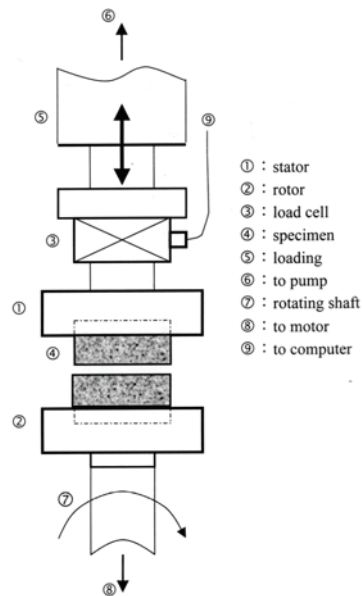


Fig. 1 Schematic diagram of wear tester.

3. Results and Discussion

3.1 Density and porosity

Fig. 2-3 shows the dependence of density and open porosity with different process parameters. Among all specimens, the preform specimen possesses the lowest density (1.21 g/cm³) and highest open porosity (42 vol %). The impregnated specimens showed lower porosity than that of preform specimen. As indicated in these figures, among impregnated specimens, pressure impregnating specimens show higher density and lower porosity than that of vacuum impregnating specimens. No matter what impregnating method used, the specimen with higher carbonization rate always exhibit lower density and higher porosity. The phenomena can be attributed to that the thermal stress induced by higher carbonization rate could possibly produce defects to increase the porosity of specimen.

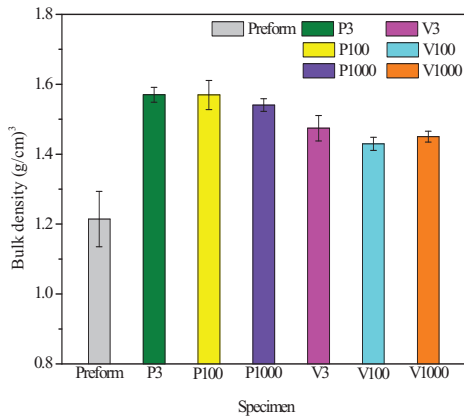


Fig.2 Bulk density of C/C specimens with different densification parameters.

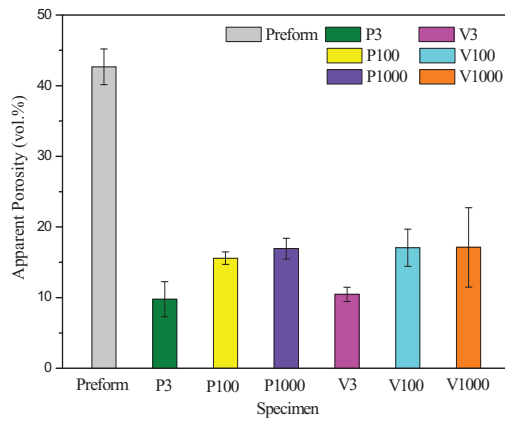


Fig. 3 Open porosity of C/C specimens with different densification parameters.

3.2 Hardness

As seen in the Fig. 4, the hardness of the preform specimen is lower than those impregnated specimens. A noticeable enhancement in hardness can be detected in impregnated specimens. As indicated in this figure, among impregnated specimens, pressure impregnating specimens show higher hardness than that of vacuum impregnating specimens. No matter what impregnating method used, the specimen with higher carbonization rate always exhibit lower hardness. The thermal stress induced by higher carbonization rate could possibly produce defects to reduce the hardness of specimen. It is noteworthy that the bad effect induced by higher carbonization rate was not so obvious for the pressure impregnating specimens. Therefore, the pressure impregnating method could mitigate the bad effect induced by higher carbonization rate.

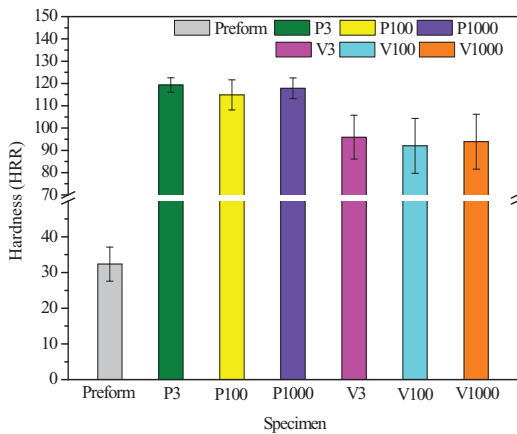


Fig.4 Hardness of C/C specimens with different densification parameters.

3.3 Friction and wear

The total average friction coefficient was taken by averaging the area integrated from the friction curve over the entire sliding interval for several runs. As seen in the Fig. 5, the total average friction coefficient of the preform specimen is lowest than all of the impregnated specimens. Fig. 2 also shows that the total average friction coefficients of those specimens with different impregnating method under same carbonization rate varied insignificantly. However, no matter what impregnating method applied those impregnated specimens with the higher carbonization rate exhibit the higher total average friction coefficient.

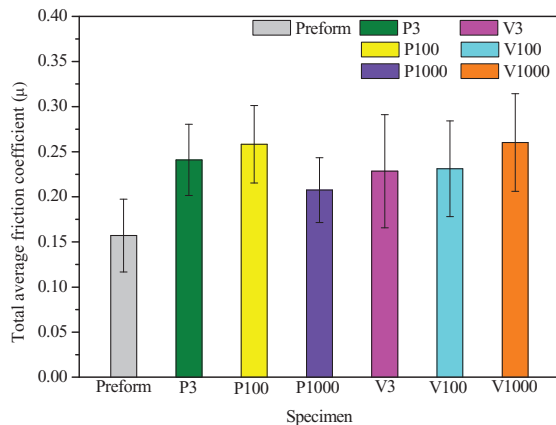


Fig.5 Total average friction of C/C specimens with different densification parameters

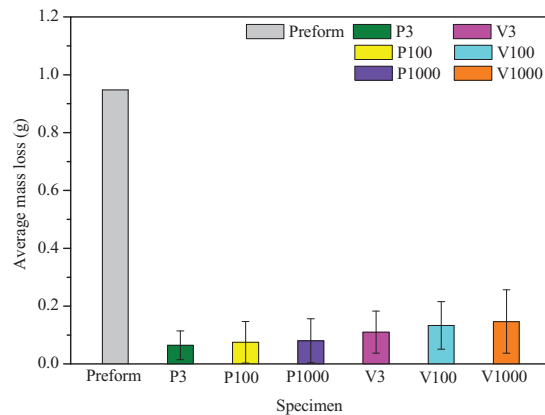


Fig.6 Mass loss of C/C specimens with different densification parameters during wear test

As seen in the Fig. 6, the preform specimen had the largest mass loss among all specimens. A noticeable reduction in mass loss can be detected in impregnated specimens. This observation implies that the impregnation process could improve the tribological performance of C/C composites. Additionally, those impregnated specimens with pressure impregnation specimens show lower mass loss than those with vacuum impregnation. It implies that the increase of infiltration efficiency could also improve the tribological performance of C/C composites. Among those specimens with the same impregnating method, the specimen with higher carbonization rate nevertheless exhibit higher mass loss. Although higher carbonization rate process could reduce the processing time and cost, some defects within specimen were then induced by the thermal stress under high carbonization rate. However, the pressure impregnating specimens with the high carbonization rate also show low mass loss in comparison with those vacuum impregnating specimen. Based on the observation of mechanical and tribological performance, it revealed that the pressure impregnating method could reduce the drawback of higher carbonization rate process and have an enormous potentiality for the C/C process.

3.4 Microstructure

Fig. 7 shows the surface morphology of C/C specimens with different densification parameters. As indicated by the arrows in Fig. 7(a), many voids or cracks resulting from the decomposition and thermal

shrinkage of polymeric matrix after carbonization can be observed in the preform specimen. These cracks and voids were believed to be the primary impregnation path for the resin impregnant to infiltrate into the specimen. No matter what impregnating method used, the impregnated specimens exhibited denser morphologies than that of preform specimen. In comparison with the vacuum impregnating specimens, the pressure impregnating specimens exhibited denser and less porous morphologies. It implies that impregnating efficiency of pressure impregnating specimens is better than that of the vacuum impregnating specimens.

In parallel, both the number and the size of cracks or voids on the surface of specimens at lower carbonization rate are found to be less and smaller than those specimens at higher carbonization rate. The thermal stress induced during the heating process is believed to cause the cracks and voids in the specimens. Therefore, as the carbonization rate increases, more defects can be formed as indicated by the arrows in the Fig 7.

Fig. 8 show the typical worn surface morphology of the specimen with different densification parameters after experiencing wear tests (0.8 MPa, 300 sec at a constant speed of 600 rpm). As shown in Fig. 8(a), the worn surface of the preform specimen exhibited a rough and random morphology. Pores, cracks and broken fibers were generated during the wear test and existed throughout the specimen. This worn surface was responsible for the large wear loss of the preform specimen during the wear process.

However, a thin smooth lubricant film and some powdery debris are found on the worn surface of pressure impregnating specimens after wearing test as shown in Fig. 8 (b)-(d). According to an earlier study [9], the worn surface with smooth lubricant film was accompanied with a relatively low friction coefficient and low wear rate. On the contrast, the worn surfaces of vacuum impregnating specimens as shown in Fig. 8(e)-(g) are mainly covered with powdery debris and pull-out or broken fibers.

Specimens carbonized at lower rate are observed to have a thin smooth lubricant film with larger area than those carbonized at higher rate. The graphene structure which forms at low carbonization rate could majorly contribute this thin lubricant film. As the carbonization rate increases; thus, more particular debris can be found on the specimens surfaces.

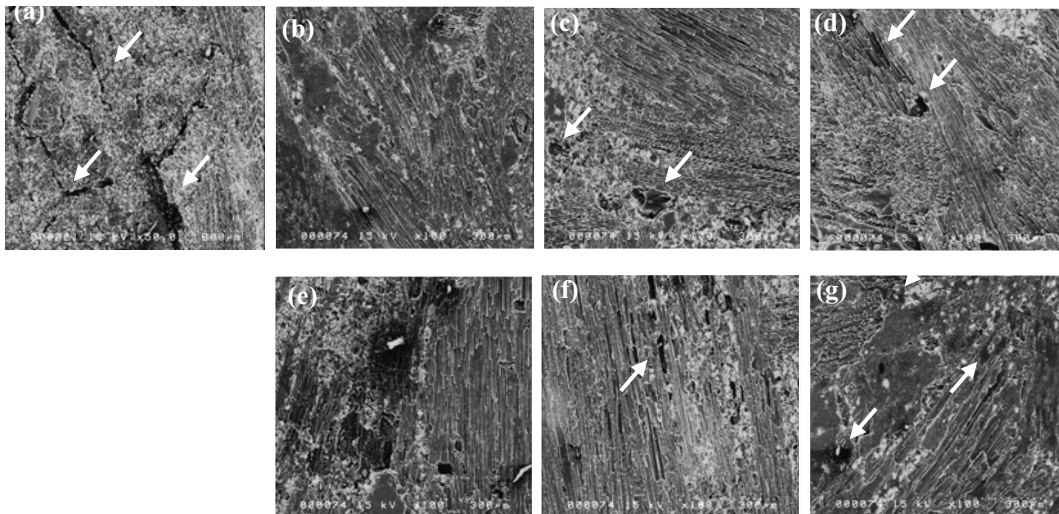


Fig.7 Surface morphologies of C/C specimens with different densification parameters. (a) preform, (b) P3, (c) P100, (d) P1000, (e) V3, (f) V100, (g) V1000.

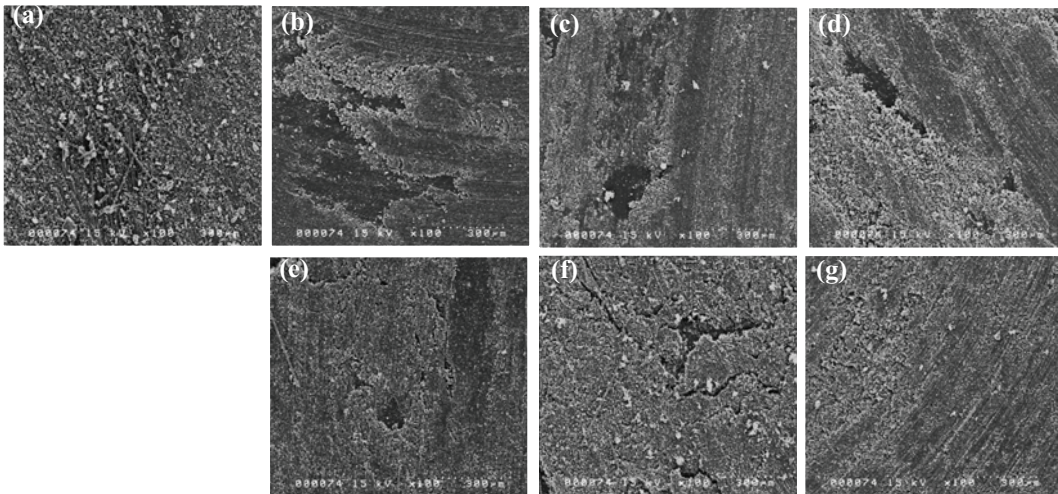


Fig.8 Worn surface morphologies of C/C specimens with different densification parameters. (a) preform, (b) P3, (c) P100, (d) P1000, (e) V3, (f) V100, (g) V1000.

4. Conclusions

Although higher carbonization rate process could reduce the processing time and cost, the specimen with higher carbonization rate exhibits worse mechanical and tribological performance. The pressure impregnating method produces a more notable effect on the properties of C/C composites than that of the carbonization rate. The pressure impregnating method collocates with higher carbonization rate process could mitigate some drawbacks of high carbonization rate process and have an enormous potentiality for the C/C process.

Acknowledgements

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References

- [1] Ruppe, J.P., *Can. Aeronau. Space J.* 1980; 26: 209-216.
- [2] Fisher R. *Composite materials in aircraft structures*, New York; John Wiley & Sons. 1990: 336-340.
- [3] Awasthi S., Wood, J.L. *Adv. Ceram. Mater* 1988; 3: 449-451.
- [4] Awasthi S., Wood, J.L. *Eng. Sci. Proc.* 1988; 1: 553-560.
- [5] Ju C.P., Lee K.J., Wu H.D., Chen C.I., *Carbon* 1994; 32(5): 971-977.
- [6] Chen J.D., Ju C.P., *Wear* 1994; 174: 129-135.
- [7] Chen J.D., Ju C.P., *Carbon* 1995; 33(1): 57-62.
- [8] Chen J.D., Chern Lin J.H., Ju C.P., *J. Mater. Sci.* 1996; 31: 1221-1229.
- [9] Chen J.D., Chern Lin J.H., Ju C.P., *Wear* 1996; 193: 38-47.

- [10] Kuo-Jung Lee, Huy –Zu Cheng, Jean-Shing Chen, *Wear* 2006; 260: 99–108
- [11] Tai N.H., Kuo H.H., Chern Lin J.H., Ju C.P., *J. Mater. Sci.* 2003; 37: 3693-3703.