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Moderation in Temperature Dependence on Counter Frictional Coefficient and Prevention of Wear of C/C Composites by Synthesizing SiC around Surface and Internal Vacancies

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Abstract**—**The aim of this study is to moderate the dependence of counter frictional coefficient on temperature between counter surfaces and to reduce the wear of C/C composites at low temperature. To modify the C/C composites, Silica (SiO_2) powders were added into phenolic resin for carbon precursor. The preform plate of the precursor of C/C composites was prepared by conventional filament winding method. The C/C composites plates were obtained by carbonizing preform plate at 2200 °C under an argon atmosphere. At that time, the silicon carbides (SiC) were synthesized around the surfaces and the internal vacancies of the C/C composites. The frictional coefficient on the counter surfaces and specific wear volumes of the C/C composites were measured by our developed frictional test machine like pin-on disk type. The XRD indicated that SiC was synthesized in the body of C/C composite fabricated by current method. The results of friction test showed that coefficient of friction of unmodified C/C composites have temperature dependence when the test condition was changed. In contrast, frictional coefficient of the C/C composite modified with SiO_2 powders was almost constant at about 0.27 when the temperature condition was changed from Room Temperature (RT) to 300 °C. The specific wear rate decreased from 25×10^{-6} mm^2/N to 0.1×10^{-6} mm^2/N. The observations of the surfaces after friction tests showed that the frictional surface of the modified C/C composites was covered with a film produced by the friction. This study found that synthesizing SiC around surface and internal vacancies of C/C composites was effective to moderate the dependence on the frictional coefficient and reduce to the abrasion of C/C composites.

Keywords**—**C/C composites, frictional coefficient, SiC, wear.

I. Introduction

CARBON fibre reinforced carbon composites (C/C composites) consist of carbon fibres and carbon matrix. C/C composites have high mechanical properties, lightness and heat-resistance [1], [2]. Because of their excellent properties, C/C composites have been used as the materials of disk brake of high-performance cars and airplanes [3], [4]. In some applications or designs for extended uses, C/C composites, unfortunately, have a problem in which the frictional coefficient was depended on the temperature condition around the frictional surface with an un-ignorable level. People who intend to utilize may well know that the frictional coefficient is low at low temperature, whereas high frictional coefficient is obtained at elevated temperature on the surfaces of C/C composites [5], [6]. For practical application to commercial vehicles, stabilization of the frictional coefficient of C/C composites has been required because frictional surface would be exposed under variable conditions of temperature. To solve the dependence of the frictional coefficient on temperature, one of the researchers produced C/C composites modified with micro sized grass fiber [7]. This method could increase the coefficient of friction at low temperature. However, the wear amount of C/C composites increased much more than commercial C/C composites disk brake.

The purpose of this study is to increase the frictional coefficient of C/C composites at low temperature to moderate the temperature dependence of it and prevent the wear. In this study, C/C composites were modified with SiO_2 powders referring the results by [8], in which a conventional sliding material was modified to decrease the wear amount by the addition of fine metal oxide powders. To make the specimens, firstly, SiO_2 powders, approximately ±12 μm, were added into phenolic resin as the material of precursor. The preform plate of the precursor containing carbon fibres as the reinforcement was prepared by conventional filament winding method. The precursor plate was prepared by curing of preform plate on a heat pressing machine. Then, the precursor was carbonized at maximum of 2,200 °C under an argon atmosphere. To measure the temperature dependence of the frictional coefficient and specific wear rate on C/C composites, friction test was carried out while test temperature was changed. The effect of addition of SiO_2 powders on the coefficient of friction, its dependence on test temperature and specific wear rate were discussed.

II. Experimental Method

A. Materials

Carbon fiber with 4,400 MPa of tensile strength and 377 GPa of Young's modulus (Torayca M40JB -6000: Toray Co., Ltd. Japan) was used as reinforcement. Phenolic resin (PR-51697: Sumitomo Bakelite Co., Ltd. Japan) was used as the precursor resin.

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B. Method for Modification of Precursor Resin

Fig. 1 shows the SiO_2 powders used in this study. To modify the C/C composite, SiO_2 powders (Marutou Co., Ltd. Japan) were utilized as the filler, in which the average diameter was approximately 12 μm. The SiO_2 powders were mixed with the phenolic resin, where the contents were 5.0 wt%, 10 wt% and 15 wt%. The modified phenolic resin was dissolved in the ethanol where the weight ratio of the phenolic resin and ethanol was 1:2. The SiO_2 powders were dispersed in the dissolved resin by a commercially available process homogenizer (L4-RT: Silverson Nippon Co., Ltd. Japan) for 30 min at 5,500 rpm.

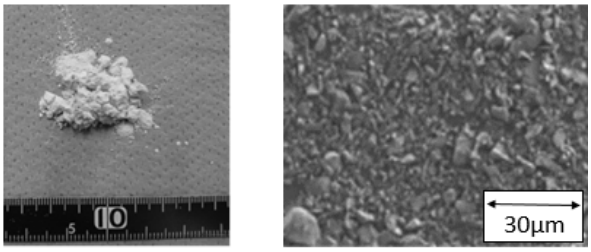

Fig. 1 SiO_2 powders in which average diameter was approximately 12 μm

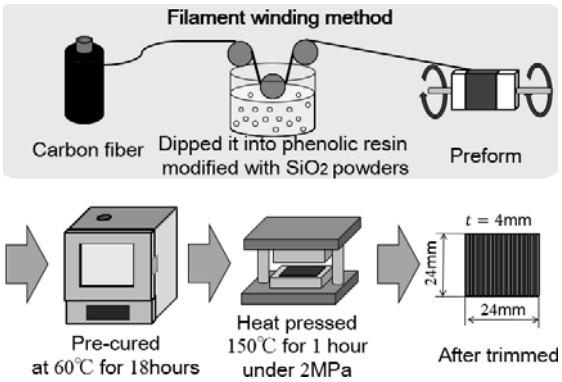

Fig. 2 Fabrication process of precursor of C/C composite

C. Fabrication of Precursor of C/C Composite

Fig. 2 shows a method for fabrication of the precursor of C/C composite. The preform plate of the precursor containing carbon fibers as the reinforcement was prepared by conventional filament winding method after dipping in the modified precursor resin as explained in the last section. The precursor plate was prepared by the curing of preform plate at 150 °C for 1 hour under 2.0 MPa on a heat pressing machine (HB200HB: Kawanaka Sangyo CO., LTD. Japan) after the pre-curing for 18 hours at 60 °C in an electric oven. After curing, the precursor plate was cut into the size of 24×24×4 mm.

D. Carbonization of Precursor of C/C Composite

Specimens of the C/C composites were obtained by through the following two processes. In first phase, the precursors were heated up to 1,000 °C at 1.39 °C/ min of the raising rate and then kept for 1 hour in the ring furnace (1-7555-18:AS ONE Corporation CO., LTD. Japan) filled with argon gas. After heating, the precursors were cooled gradually. In second phase, specimens were heated up to 2,200 °C at 22.0 °C/min of the raising rate and then kept for 1 hour in the carbonize furnace filled with argon gas. After heating, the precursors were cooled gradually.

E. Friction Test

Fig. 3 shows the schematic illustration of our frictional test machine like a pin-on disk type, where the temperature condition was changed from RT to 100, 200 and 300 °C. In this test, frictional force was applied between fixed quadratic specimen (which had 7×7 mm of square cross section and 4 mm thickness) and rotational disk specimen (which had 24×24 mm of square cross section and 4 mm thickness), where constant revolution was kept in a same axis. The applied normal load was 49 N by weight to the fixed specimen. The number of revolutions of the AC motor was controlled to be 300 rpm. The resultant torque and the normal force between the two counter specimens were monitored by a load-cell and torque meter, respectively, in order to determine the coefficient of friction. The coefficient of kinematic friction μ was calculated by;

$$\mu = \frac{T}{Nr_{eff}} [-] \quad (1)$$

where μ, T, N and r_{eff} denote the coefficient of kinematic friction, the torque, the normal force and effective radius, respectively.

The specific wear rate W was calculated by;

$$W = \frac{V}{Nl} [\mathrm{mm^2/N}] \quad (2)$$

where W, V, N and l denote the specific wear rate, worn volume of test piece, the applied normal force and the sliding distance.

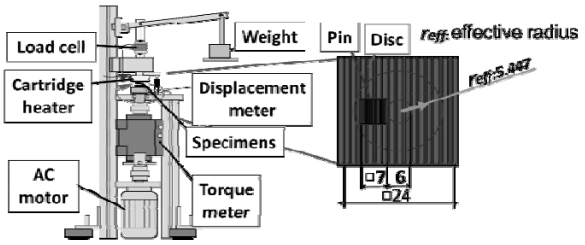

Fig. 3 Illustration of rotational friction test machine

III. Result And Discussion

A. XRD Analysis of C/C Composites

Fig. 4 shows the result of X-ray diffraction (XRD) patterns of the surface of C/C composites modified with SiO_2 powders. The result of XRD shows that the materials was highly crystallized and composed mostly of β-SiC. In [8], the β-SiC is synthesized when graphite is exposed to SiO gas at over 1973 K of temperature. The current result suggested that added SiO_2 powders would be gasified over 1973 K and generate SiO gas as;

$$SiO_2(s) + 3C(s) = SiO(g) + CO(g)$$

A part of the SiO gas reacted with carbon to produce β-SiC as;

$$SiO(g) + 3CO(g) = SiC(s) + 2CO_2(g)$$

The XRD observation in the current study indicated that SiC was successively synthesized in the body of fabricated C/C composites by the current method.

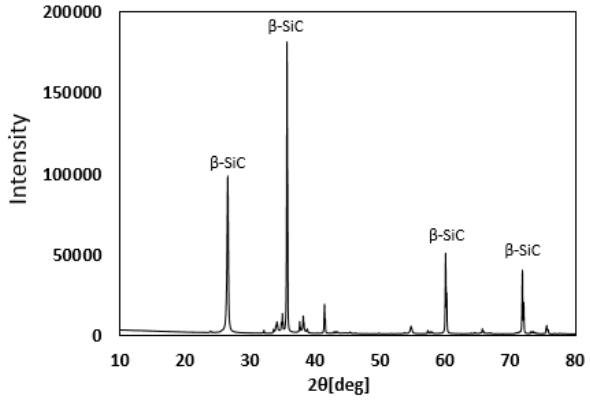

Fig. 4 X-ray diffraction of C/C composites modified with SiO_2 powders

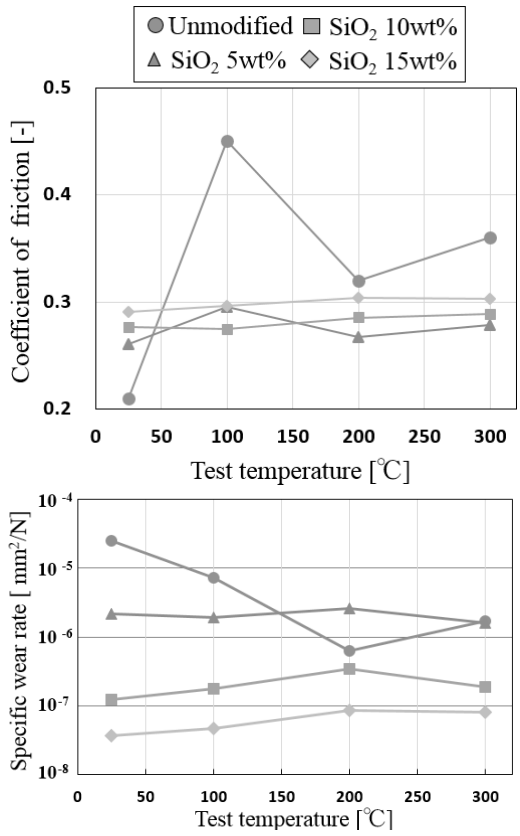

Fig. 5 Coefficient of friction and specific wear rate with respect to the test temperature

B. Coefficient of Friction and Specific Wear Rate of C/C Composites

Fig. 5 shows the change of coefficient of friction and specific wear rate of C/C composites with respect to the test temperature. The result showed that the coefficient of friction of unmodified C/C composites was varied from about 0.21 to 0.45 when the temperature condition was changed from RT to 300 °C. In contrast, the frictional coefficient of the modified C/C composites was almost constant at about 0.27 even if test temperature condition was changed. The change range of coefficient of friction ($\Delta\mu=\mu_{max}-\mu_{min}$) with respect to the test temperature was decreased from about 0.24 to 0.013 by the addition of 15 wt% of SiO_2 powders into C/C composites. The specific wear rate was also decreased from about 25×10^{-6} mm^2/N to 0.039×10^{-6} mm^2/N by the addition of 15 wt% of SiO_2 powders into C/C composites at RT. The change range of specific wear rate with respect to the test temperature ($\Delta W=W_{max}-W_{min}$) was decreased from about 6.6×10^{-6} mm^2/N to 0.048×10^{-6} mm^2/N by the modification. The maximum improvement in the change range of coefficient of friction and the specific wear rate were obtained when the content ratio of the SiO_2 powders was 15wt%.

C. Worn Surface Observations

Fig. 6 shows surface states on the specimens observed by a laser microscope after friction test. The observation of the surface of unmodified specimen at RT showed that the rough surface was observed and fracture of fibers was occurred, indicating that the loss of material was caused by the fiber fragmentations of carbon fibers. In contrast, it was observed that particles attaching to the surface and the sizes of particles were relatively small compared with those at 300 °C. The coefficient of friction of unmodified C/C composites was increased by formation of thin film by rolling of tiny particles at high temperature. In contrast, the worn surface of C/C composites modified with SiO_2 powders was covered with a smooth film and some scratch marks were observed, even at low temperature condition as well as at high temperature condition. These results suggested that friction mode and wear mechanism were changed by the addition of SiO_2 powder into C/C composites.

Fig. 7 shows the result of the Energy Dispersive X-ray Spectroscopy (EDS) analysis of surface on addition of SiO_2 powder into C/C composites after friction test at RT and 300 °C. The EDS observation indicated that SiC particles were partly distributed on the surface after the friction test, and it was confirmed that crystals of SiC having the same size as the size of scratch mark were observed on the surface after the friction test. These results suggested that abrasive friction was occurred because of synthesizing SiC around the surface and the internal vacancies. Fig. 8 shows the SEM and EDS images of the surface state on the specimens on addition of 5, 10 and 15wt% of SiO_2 powders with respect to the temperature. The SEM and EDS observations indicated that smooth friction film was observed on the worn surface, when content ratio of SiO_2 powders increased, and number of SiC particles was increased. Because of these results, the specific wear rate was decreased

when content ratio of SiO_2 powders increased.

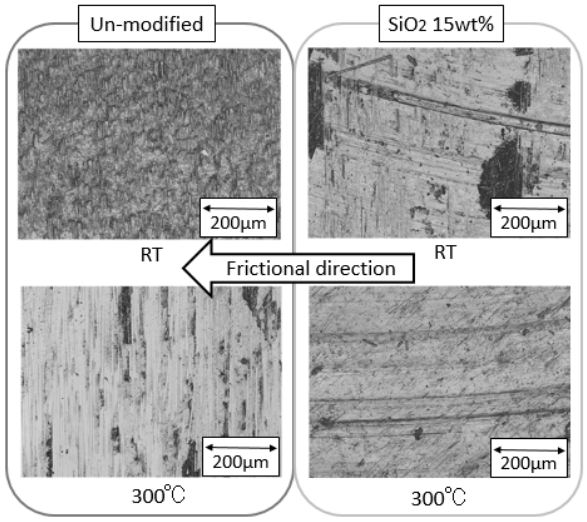

Fig. 6 Worn surface of C/C composites after friction tests

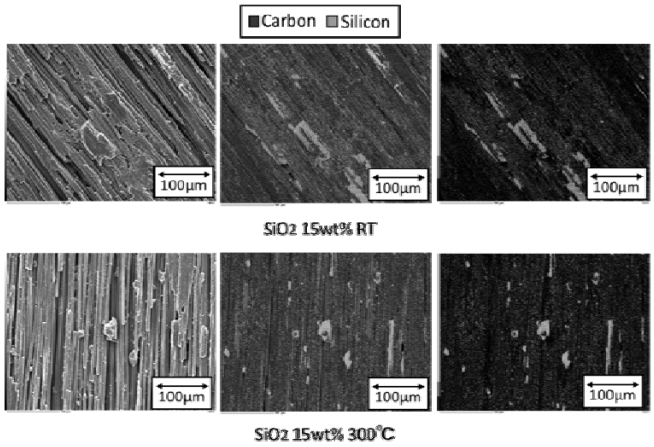

Fig. 7 EDS images of after friction tests

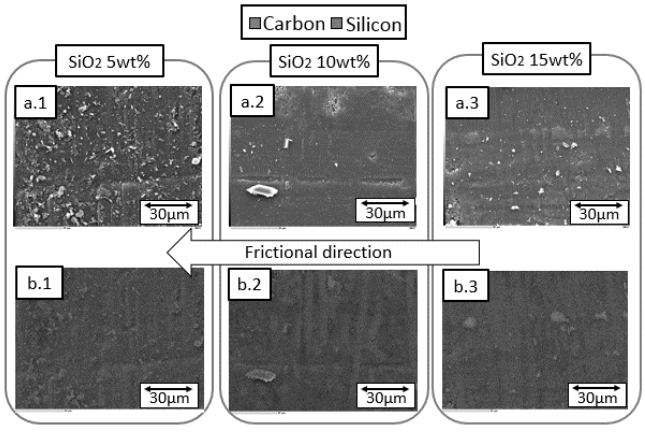

Fig. 8 Worn surface of C/C composites addition of 5, 10 and 15wt% of SiO_2 powders (a) SEM images, (b) EDS images

IV. Conclusion

In this study, effect of modification with SiO_2 powders on dependence of coefficient of friction and spcific wear rate between C/C composites on temperature condition was investigated. The following conclusions were given.

1. The XRD observation in current study indicated that SiC was successively synthesized in the body of fabricated C/C composites by current method.
2. The coefficient of friction of the specimen modified with SiO_2 powders was almost constant with respect to the temperature.
3. The specific wear rate was decreased by the addition of SiO_2 powders into C/C composites.
4. Due to the synthetization around the surface and the internal vacancies of C/C composites by addition of SiO_2 powders into carbon precursor, the frictional coefficient was increased by the scratching with exposed particles of SiC the friction surface, and specific wear rate was decreased by the friction film of worn surface was smoother because of synthesizing SiC around the surface and the internal vacancies of C/C composites.

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