

Sliding Behavior of C/C and C/C-SiC Composites in Acidic Environment-Against High Chromium Steel Disc

V. K. Srivastava

Department of Mechanical Engineering, Institute of Technology, BHU, Varanasi, 221005, India

Abstract The wear behaviour of carbon-carbon (C/C) and carbon-carbon-silicon-carbide (C/C-SiC) composites were studied using a pin-on-disc test rig under dry condition before and after immersion in 25% concentrated H₂SO₄. The influence of the weight loss and worn temperature against sliding distance and time were investigated. It was found that the weight loss increases with increasing of sliding distance and time, where worn surface temperature increases slowly up to 1.5 Km sliding distance and then tended to become constant. Also, weight loss and worn surface temperature reduces with the exposure of 25% concentrated sulphuric acid. However, the C/C-SiC composites exhibiting a lower weight loss but higher worn surface temperature compare to C/C composites at constant load. The wear and worn temperature of C/C composite gradually increases compare to C/C-SiC composite, with an average fading ratio of 7.7% (C/C composite) and 7.4% (C/C-SiC composite). Scanning electron microscope and projection microscope micrographs show that the graphite film was transferred on the test pin side, which stabilized the increase in test pin temperature after the formation of a thick film of graphite debris.

Keywords Sliding Wear, Carbon-Carbon Composite, Carbon-Carbon-Silicon-Carbide Composite, Mild Steel, Scanning Electron Microscopy

1. Introduction

Carbon/carbon (C/C) composites have received considerable attention in the aircraft and automobile brake industry as strong candidate materials because of their low density and unique thermal and mechanical properties[1-3]. In order to utilize these materials as friction materials, the evaluation of their wear behavior is important. However, very little effort has been made to characterize the wear behavior of C/C composites. Recently some researchers have studied the wear of C/C composites under representative aircraft braking conditions[4-7]. They have found that oxidation in the subsurface was the predominant wear mechanism due to the high interfacial temperature of the friction material and, as a result, most previous works have concentrated on the oxidative mass loss of C/C composites. Under braking conditions the material loss of the friction surface also occurs with the oxidative mass loss in the subsurface. Therefore, the investigation of frictional wear is also important to characterize the wear behavior of C/C composites. However, the frictional wear mechanism of C/C composites is not understood well at this stage.

C/C-SiC composites, made by liquid silicon infiltration (LSI-process), offer superior tribological properties in terms

of high coefficient of friction and wear resistance. The carbon fibres lead to an improved damage tolerance in comparison to monolithic SiC, whereas the silicon carbide matrix improves the wear resistance compared to carbon/carbon (C/C) composites. C/C-SiC composites are therefore new, outstanding materials for brakes and clutches of high speed cars, trains and emergency brakes in the field of mechanical engineering and conveying.

First attempts to investigate C/C-SiC composites for their use as frictional materials for brake pads and disks started in the early nineties[8]. C/C-SiC material shows, in comparison to carbon-carbon, a considerably lower open porosity (less than 5%), a moderately higher density (about 2g/cm³) and a ceramic share of at least 20% in mass. Several activities in institutes as well as in industries now exist to investigate CMC materials for their use as frictional materials for brake pads and disks[9-12]. The resulting materials differ in their constituent (fibres, fillers), microstructure (ceramic content, density, strength, and thermal conductivity) and also in their processing conditions (fibre coating, temperature, etc.). Nevertheless, they are all based on carbon fibres and silicon carbide matrices as the main constituents of the composite materials. The carbon fibres generally decrease the brittleness of SiC considerably so that the damage tolerance of C/C-SiC components lies in the same order of magnitude as for grey cast iron.

In this study the wear behavior of C/C and C/C-SiC composites were investigated against sliding on high chromium steel disc before and after exposure of 25% concen-

* Corresponding author:

vijayks210@gmail.com (V. K. Srivastava)

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trated sulphuric acid. Weight loss and worn surface temperature were measured with the variation of sliding distance and time at constant load of 6 Kg. Finally, micrographic analysis was used to obtain detailed information about the worn surfaces of the composites.

2. Experimental Procedure

2.1. Materials and Specimen

The materials used in this investigation were two kinds of cross-woven C/C and C/C-SiC composites. The C/C-SiC composite was manufactured by liquid siliconising infiltration (LSI) method. Usually, the LSI-process gives C/C-SiC composites containing significant amount of unreacted free carbon together with unreacted free silicon. C/C-SiC ceramics were oxidized in ambient air/oxygen. However, the temperature to which the samples were subjected and the holding times play an important role. C/C-SiC was fabricated in the form of rectangular plates of 3 mm nominal thickness. C/C-SiC is a tough ceramics. The investigations refer to cross-woven (0°/90°) C/C-SiC composites with high tenacity (HTA) carbon fibre ($\phi=7\mu\text{m}$) as reinforcement, and the matrix was silicon carbide. The volume fractions are 60% carbon fibre, 38% silicon carbide and 2% silicon.

The carbon/carbon (C/C) composite was produced by processing route of yarn method with 3 mm thickness, fibre orientation of 0°/90° and 50 % volume fraction. Polyacrylonitrile (PAN) based high modulus (HTA) carbon fibre (Torayca M40) was used because of good properties at high temperature.

The plates were cut into the desired shape (cuboids 25 mm x 3 mm) for the wear test. All the wear test specimens were polished to a surface roughness of about 0.3 μm . Before wear testing, the composite test pins and steel disc counterpart were cleaned with carbon tetrachloride and dried. Some specimens were immersed in the 25% concentrated sulphuric acid for the fixed period of time 312 hrs.

2.2. Wear and Temperature Tests

The apparatus used in this investigation was a pin-on-disc type apparatus. A test pin (3 mm x 3 mm cross-section and 25 mm length) was inserted into the hole of the specimen holder. The outer diameter was 8 mm, the length 36 mm, with a rectangular 4 mm x 4 mm x 20 mm blind hole, designed for holding the test pin. A clamping device (screw) was provided on the holder for holding the test pin. To avoid vibration on the test pin, a mild steel strip (0.5 mm thick x 3 mm wide and 20 mm long) was inserted with the test pin. A high-carbon chromium steel disc of 120 mm diameter, heat-treated to a hardness of 63 HRC, was rigidly mounted on a brass disc to avoid vibration of the motor. The disc was also polished with a fine grade of emery paper, followed by cleaning with carbon tetrachloride solvent, and dried in air. The weight platform was directly attached to the one end of the test pin, and the second end of the test pin was just

touching the chromium steel disc for the measurement of wear. The test specimen was directly loaded with constant weight 6 Kg (58.8 N). The wear losses were recorded by an electric balance to an accuracy of ± 0.1 mg. The difference in weight of the test pins before and after the experiment was the weight loss of the sample. The position of the test pin over the disc was changed to produce wear tracks of various diameters. A new pin and fresh wear track were adjusted by regulating the speed of the variable direct-current motor. The sliding speed and time at different tracks were also changed for the measurement of wear loss. The wear loss of C/C and C/C-SiC composites specimens were measured before and after exposure of 25% concentrated sulphuric acid.

A digital thermocouple was used to measure the temperature of the worn surface. The rise in temperature was noted at intervals of 30 min. The same procedure was adopted at various sliding speed and times. The fading ratio D of the worn surfaces in moist condition, which represents the sensitivity of the worn surface temperature to an acidic condition, can be determined according to the following relation;

$$D = (1 - T_{\text{acid}} / T_{\text{dry}}) \times 100\% \quad (1)$$

where, T_{acid} and T_{dry} correspond to the worn surface temperature in acidic and dry condition respectively. The fading ratio of C/C and C/C-SiC composites was obtained with the variation of sliding speed.

Finally, a scanning electron microscope (JEOL-840) and projection microscope were used for the morphological analysis of the sliding surfaces and debris from the test pin.

3. Results and Discussion

Figure 1 and 2 shows the variation in the weight loss as a function of sliding speed and sliding time for C/C and C/C-SiC composites under dry and acidic condition. The weight losses increased with increase of sliding distance and time, tended to become steady after 3.0 Km.

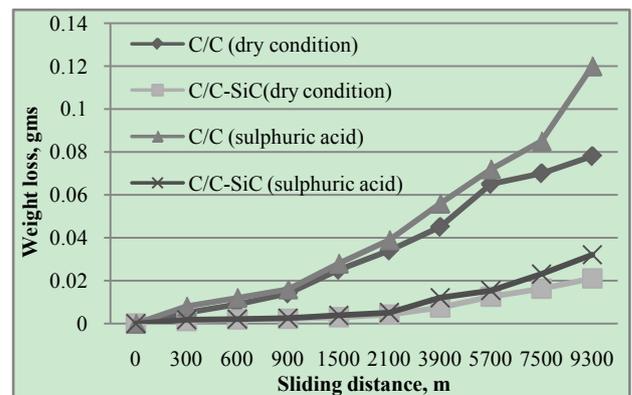


Figure 1. Variation of weight loss with sliding distance at constant load of 6Kg of C/C and C/C-SiC composites under dry and sulphuric acid condition

The results show a similar trend for C/C and C/C-SiC composites under dry and acidic environment. The weight losses of C/C composite are higher than the C/C-SiC composite. This indicates that the graphite peeled off and spread

between the mating surfaces of the test sample and disc. Initially, the weight loss was lower before 1.5 Km, then after, weight loss increased very rapidly and tended towards steady state. Lower weight losses indicate that the fibre/matrix bond was stronger.

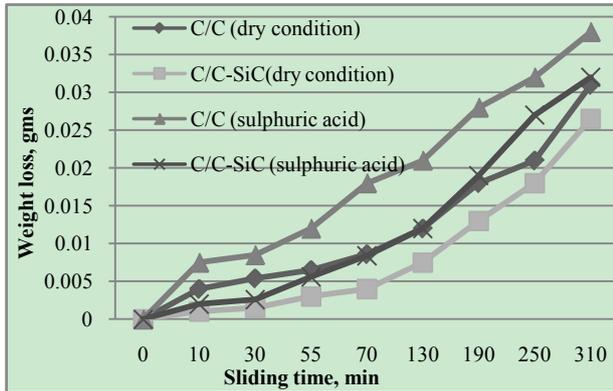


Figure 2. Variation of weight loss with sliding time at constant load of 6 Kg of C/C and C/C-SiC composites under dry and sulphuric acid condition

The result clearly indicates that a transition occurred in all samples somewhere between 7.5 and 8.5 Km. When the transition occurred, the weight losses of the specimen tend to decreased. After the transition period, the worn temperature of the counter face stabilized, as shown in Fig. 3.

From these results it is considered that the sliding speed and time are affected identically to the wearing behavior of C/C and C/C-SiC composites. However, it is cleared from the results that C/C-SiC composite turned higher worn surface temperature than the C/C composite, because C/C-SiC composite contained hard silicon particles, which resulted higher temperature against the sliding on high chromium steel disc[5]. The worn temperature of C/C composite is tended to steady state after sliding at 1.5 Km. But, C/C-SiC composite is becomes steady state after the sliding of 3.9 Km.

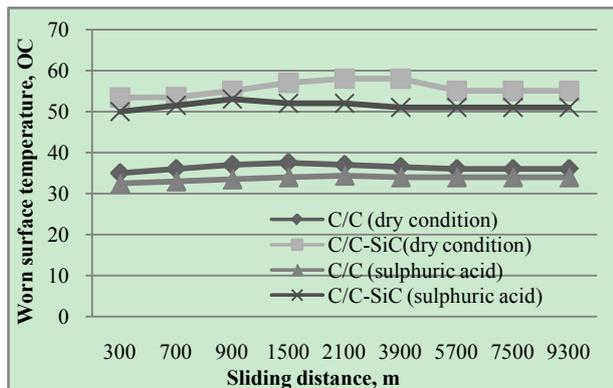


Figure 3. Variation of worn surface temperature with sliding distance at constant load of 6 Kg of C/C and C/C-SiC composites under dry and sulphuric acid condition

Fig. 4 shows that fading ratio of C/C composites increased very rapidly and gradually with increase of sliding speed. Whereas fading ratio of C/C-SiC composites increases abruptly with increase of sliding speed. This clearly indicates that the C/C composite losses more debris compare to

C/C-SiC composite. It is observed that breakage and loss of fibres occur significantly and the loose particles at the contact interface started abrading the surface together with the shearing. These abrading particles gave rise to more loose particles, and in this way a higher surface wear rate began in a cumulative fashion[5]. The matrix tore out and the surface was covered with graphite powder as shown in Figs. 5 and 6. Large worn particles were formed (Fig. 5), leading to high rates of wear.

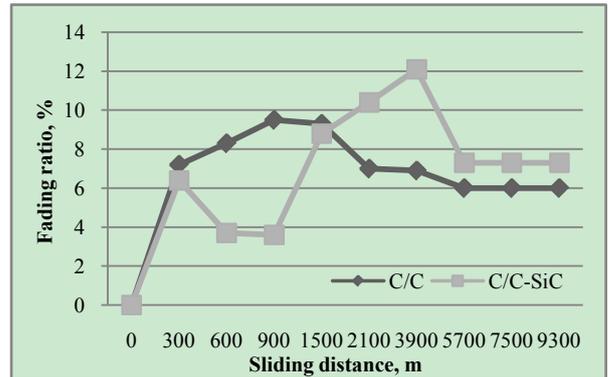


Figure 4. Variation of fading ratio with sliding distance at constant load of 6 Kg of C/C and C/C-SiC composites

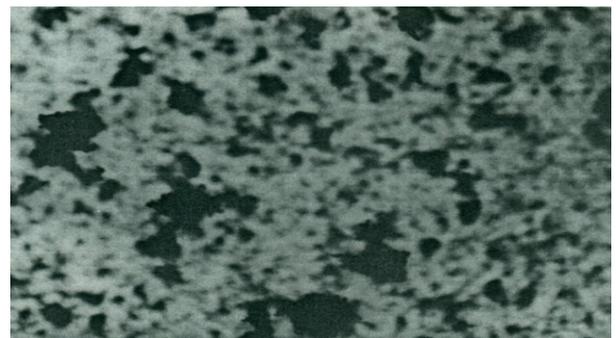


Figure 5. Projection micrograph (100X) showing thick steady state debris of C/C composite under dry condition at load of 6 Kg and sliding speed 0.5 m/s

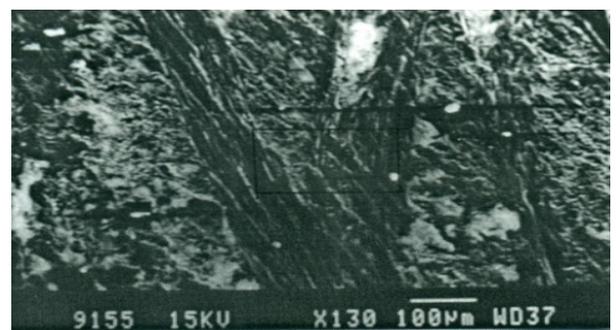


Figure 6. SEM micrograph shows the effect of wear on counterpart of C/C composite (exposed with sulphuric acid) at the load of 6 Kg and sliding speed 0.5 m/s

Fibre/matrix interfaces were debonded, and deep cracks initiated and propagated along the longitudinal as well as transverse fibre directions, as shown in Fig. 6. Silicon carbide particles also come out from the matrix and are even dislodged from the surface, which reduced the formation of debris on the counter side, as shown in Fig. 7.

Some of the worn debris remains on the worn surface of the tested C/C and C/C-SiC composites during breaking with a few worn debris adhered to the corresponding steel disc. A magnified view of the micrographs shows clear microcrack generation along the longitudinal fibre direction (Fig. 8).

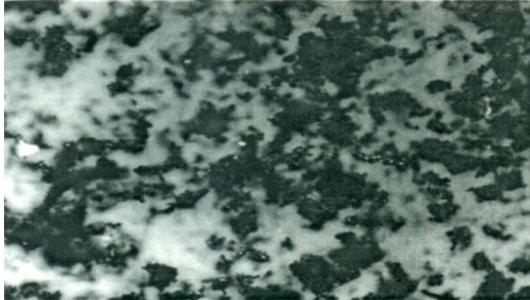


Figure 7. Projection micrograph (100X) showing steady state debris of C/C-SiC composite under dry condition at load of 6 Kg and sliding speed 0.5 m/s

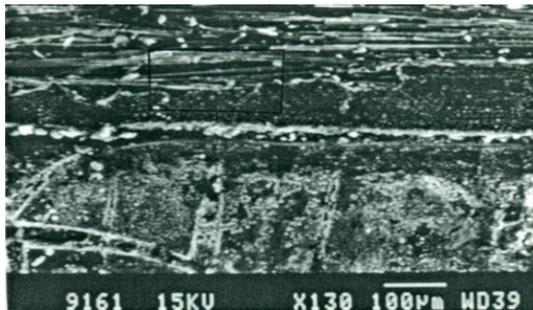


Figure 8. SEM micrograph shows the effect of wear on counterpart of C/C-SiC composite (exposed with sulphuric acid) at the load of 6 Kg and sliding speed 0.5 m/s

In this case, thinning breakage and removal of those fibres embedded longitudinally on the surface occurred, as can be seen from Figs. 6 and 8. Fibre-matrix debonding also dominated due to high mechanical stresses. SEM micrographs of the worn debris of H_2SO_4 exposed composites are appearing to be a typical characteristic of abrasive wear. The C/C-SiC composites have many micro convex bodies on the worn surface, including hard SiC phase, resin carbon, and second hard phase Si. These hard particles cause the increase of the worn surface temperature during the sliding times. Additionally, the local surface of C/C-SiC composites adheres to the corresponding steel disc easily due to the lack of self lubricants. On the other side, C/C composites tended to self lubricants because of thick film of graphite particles. At the same time, Fe on Cr steel disc reacts with resin carbon or graphite under the condition of higher external stress and temperature[12]. However, it is cleared that 25% concentrated sulphuric acid affected very marginally on the wear behavior of C/C and C/C-SiC composites. Under exposed condition of C/C-SiC composite with H_2SO_4 , SiC matrix reacts with adsorbing moisture that evaporates under friction heat and produces H_2SiO_3 and Fe has chemical reaction with water and forms $Fe(OH)_3$ as well[12]. Similarly, under exposed condition of C/C composites H_2SO_4 , C matrix reacts with adsorbing moisture that also evaporates under friction heat and produces SO_2 and CO_2 . Therefore, the weight loss

increases very slowly in dry condition comparison to acidic condition. It confine that the composite is very much affected in the sulphuric acid which losses the interface bond strength because sulphuric acid swell the matrix and weaken the debonding strength of fibre and matrix. Also, it is observed that the very sharp cracks nucleate and propagate through the interface bond due to direct weakening of the carbon fibre and matrix by the acid. Therefore, the formation of a zone of bridged fibres, matrix cracking, swelling and matrix diffusion are more pronounced with the exposure of acid on C/C and C/C-SiC composites[6]. Considerable debris was transferred on the sliding surfaces and tip of pin. The size of debris particles depended on the sliding distance and time. However, when the composite debris was abraded for a longer duration, it formed smooth, dense and bright debris. The black debris indicates that particles are graphite powder, which acts as a self lubricant[7,8].

4. Conclusions

Experiments were carried out to study the effect of 25% sulphuric acid on the wear behavior of C/C and C/C-SiC composites with the variation of sliding speed and time. Based on these studies, following conclusions are drawn:

*For a particular load, the wear of C/C and C/C-SiC composites increased with increase of sliding distance / time and tended to steady state, which indicates that the sliding temperature increased and attained a steady state after the transfer of thick graphite film to the counter face, which acts as a self-lubricating film.

*The wear behavior of C/C and C/C-SiC composites marginally affected by the exposure of 25% sulphuric acid for the period of 312 hrs.

*The fading ratio of C/C composite increased very rapidly with increase of sliding distance and appeared to steady state after 1.5 Km sliding distance, whereas, fading ration of C/C-SiC composite turned random behavior with increase of sliding distance.

The C/C composites resulted higher wear losses compare to C/C-SiC composites during the sliding against chromium steel disc. Micrographic studied show that the thick film of debris on the counter sides transferred in the case of C/C composite compare to C/C-SiC composite.

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