Effect of Carbon Fiber Addition on Ceramic Reinforced Phenolic Resin Based Friction Composites

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Abstract

Carbon fibers have been used as additional reinforcing fibers to improve the mechanical and tribological properties of phenolic resin-based ceramic-carbon composites. The composites comprising ceramic particulates such as Silicon carbide, Boron carbide of 1-30 micron size as reinforcement and phenolic resin as matrix carbon precursor were prepared by compaction method followed by carbonization to 1000°C in inert atmosphere. Experimental results indicate that carbonization results in decrease in thickness and weight, the amount of reduction increasing with addition of carbon fibers results in compact high density composites. Composites comprising of 10 wt. % fibers exhibited maximum hardness, compressive strength and density after carbonization. Tribological properties of the composites were evaluated against Cr6 ball using a pin-on-disc Tribometer with different linear speed, sliding distance and load conditions. It was found that the composites filled with lower amount of carbon fibers showed relatively higher friction coefficient value. Also, it was noted that friction coefficient increases with increase in the applied load (1N, 2N and 5N) and linear speed.

Introduction

Friction based materials usually consist of several ingredients which are classified as binders, fillers, abrasives and friction modifiers [1, 2]. Friction materials generally refer to brake linings and clutch facings. Brake linings are used especially in transportation, automotive, aircraft, railroad and trucking industries. Other heavy users of brake linings are drilling rigs and constructions. Friction materials can be classified as organic (polymeric), carbon-based, asbestos-based, metallic and phenolic based materials [3]. Organic based friction materials are usually processed through hot-pressing. Ten to twenty different compositions are mixed in which each component imparting their characteristic property. Metallic fibers/carbon fibers were used to increase strength and stability while for lubrication and stabilization of friction materials graphite or metallic sulfides are considered. To reduce the production cost and the binding property silicon based oxide/ barium sulphate and polymeric resin/ elastomers are added [4]. Carbon based materials (Carbon/Carbon friction materials) are composed of woven carbon fiber with a carbon binder produced by chemical vapor deposition (CVD) of hydrocarbon gas or pyrolysis of thermosetting resins/thermoplastics pitches. Carbon binder not only holds the fibers together but also offers excellent mechanical and tribological properties. Asbestos based friction materials used to be either molded or woven but are no longer preferred due to release of fine dust during usage which are hazardous to environment. Metallic and Semi-Metallic friction materials were first seen as high-performance alternative to asbestos materials. Metallic based friction material consists of steel fiber, iron powder, low cost carbon powder, phenolic resin and clay and organic fillers. Phenolic resin-based friction materials are widely used in automobile and aviation industries, due to their high specific strength, low density, and good cost-effectiveness of raw materials. However, the tribological application of phenolic resin-based friction materials is usually limited owing to the relatively poor stability and wear resistance. Therefore, it is imperative to incorporate various reinforcing and filling constituents such as fillers (Particulates and fibers) and friction modifiers (solid lubricants) into phenolic resin-based friction composites for the purpose of increasing the mechanical strength and friction property [5–8].

Fillers used in the form of particulates are usually added to phenolic resin-based friction materials to improve their stiffness and strength. Fibers can be

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added as second phase filler material. This second phase filler material will influence the friction property of composite material. Fiber reinforcements, *e.g.*, carbon, glass, and aramid fibers, are the main candidates and have been widely employed nowadays. Carbon fiber reinforcement dominates in high-performance applications as compared to other fibers because of its outstanding mechanical properties combined with low weight and high thermal stability. With the aim of using fiber as filler, S.C. Ho et al [9] investigated the use of different fibers, viz. cellulose, steel, ceramic, carbon, brass and copper fibers on mechanical and tribological properties of copper/phenolic resin composites. Results indicated except cellulose, all fiber-added materials exhibit higher average COF values as compared to materials having no fibers. Steel fiber-added material has the largest wear, while copper fiber-added material has the smallest wear. Du-Qing Cheng et al [10] studied new composite materials for brake applications which were fabricated using metallic powders, barium sulphate and modified phenolic resin as the matrix and carbon fiber as the reinforced material. This was compared with glass fiber and asbestos reinforced composites with same matrix for comparison. Carbon fiber-reinforced friction materials showed lower wear rate than those of glass fiber and asbestos fiber-reinforced composites in the temperature range of 100°C–300°C. It is interesting to see that the friction coefficient of the carbon fiber-reinforced friction materials increases as frictional temperature increases from 100°C to 300°C, while the frictional coefficients of the other two composites decrease with temperatures. Tanaka et al. [11] proposed a material comprising 5–20 wt% binder resin, 10–50 wt% carbon or aromatic polyamide fiber, 5–30 wt% solid lubricant and 5–20 wt% ceramic powder depicting such advantages as high and stable friction force along with excellent wear resistance. Composite with modified resin and appropriate hardener content have been reported to prove better than the composites with traditional phenolic in all the Tribo-performance properties including performance μ (coefficient of friction), fade μ (coefficient of friction), disc temperature rise and wear [12, 13]. P. Fourneir et.al. [14] studied the tribological behavior of C - SiC composites reinforced with additional material (carbon fiber) under different experimental conditions (velocity, load and temperature). The experimental data shows that the addition of carbon fibers permits the tribological performance of these materials to be increased. P Xiao [15] et. al fabricated carbon fiber reinforced carbon and SiC dual matrices composites by warm compacted-in situ reaction. The C/C-SiC brake composites showed excellent tribological performance, including high coefficient of friction (0.38), good abrasive resistance and brake steadily on dry condition.

It seems obvious from the above observations that the addition of carbon fibers as additional filler could impose the mechanical and tribological behavior of ceramic reinforced phenolic resin based composites. However, the studies mentioned above either contain different compositions or use different processes. The purpose of the present work was thus to study the effect of addition of carbon fibers on the mechanical and tribological properties of carbon-ceramic composites with ceramic powders such as SiC and B_4C as powder ceramic reinforcements and phenolic resin derived carbon as matrix.

Experimental

Raw materials

Phenolic resin mixed with 12 % hardener (Novolac type) manufactured by Polymers and Polyols Chemicals, Vapi was used as the matrix material. Two types of hard ceramic particles, viz. Silicon carbide and Boron carbide manufactured by Carborundum, Thiruvanathpuram, and Boron carbide Pvt. Limited, Navasari respectively were used as particulate fillers. Carbon fibers (PC 19) processed from polyacrylonitrile based polymer was used as second phase filler which were chopped into micron size to attain proper mixing during the processing of composites. The density of these fibers, obtained from float and sink method was 1690 kg/m^3. The compositions of the fabricated composites are provided in Table 1. The particle size distribution was measured using a sieve shaker which is presented below in Table 2.

Table 1

Compositions of the fabricated composite materials

Raw Materials	(wt. %)
Phenolic resin	30-50%
Calcined Petroleum coke	20-30%
SiC	10-30%
B_4C	10-30%
Carbon fiber	0-10%

Table 2
Particle Size Analysis of different constituents used in the development of ceramic reinforced phenolic resin based friction

Constituents	Particle size (microns)
Phenolic resin	10-100 microns
Calcined petroleum	Micron Size
Silicon Carbide	10-20 microns
Boron carbide	1-1.5 microns

Methodology

Fiber-free and fiber-added ceramic reinforced phenolic resin-based friction composites used for this study were prepared by dry mixing appropriate amounts of 150 mesh-sized phenolic resin powder and ceramic particles. Carbon fiber content was varied from 0-10 wt%. Composites containing higher carbon fiber content were difficult to mix during ball-milling due to which significant clustering and entanglement were observed. Therefore, milling time was enhanced as the percentage of carbon fiber was increased. This was followed by hot pressing the mixture in a 150 mm×30 mm×5-6 mm HCHC die by simultaneously heating to 150°C for 30 minutes under a pressure of 19.6 MPa (200 kg/cm^2). Green compacted composites were then carbonized in inert atmosphere at 1000°C with slow heating/ cooling rates. These composites were characterized for physical, mechanical and tribological properties. A schematic diagram of processing of ceramic reinforced phenolic resin based composites is shown in Fig 1.

Characterization of Composites

Weight loss and reduction in thickness of each sample were measured using a triple-digit electronic balance and a digital verneir caliper (Mitutoyo, Japan), respectively. Density and porosity of composites were calculated according to the ASTM C-838-91 and ASTM C-20-92 respectively. Hardness and Compressive tests were conducted on the fabricated specimens. Rockwell Hardness values were determined using a hardened steel ball penetrator with a diameter of ¼ inch under a major load of 60 kgf and minor load of 10 kgf (HRL). Bond strength tests were carried out on Instron 5500 machine having a 150 KN load cell. Crosshead displacement velocities of 0.5 mm/min were maintained for compressive testing. Specimen dimensions for the compressive testing were 25×5×5^{-6} mm^3. Crosshead displacement was used to calculate strain in the compression testing. Load and displacement data

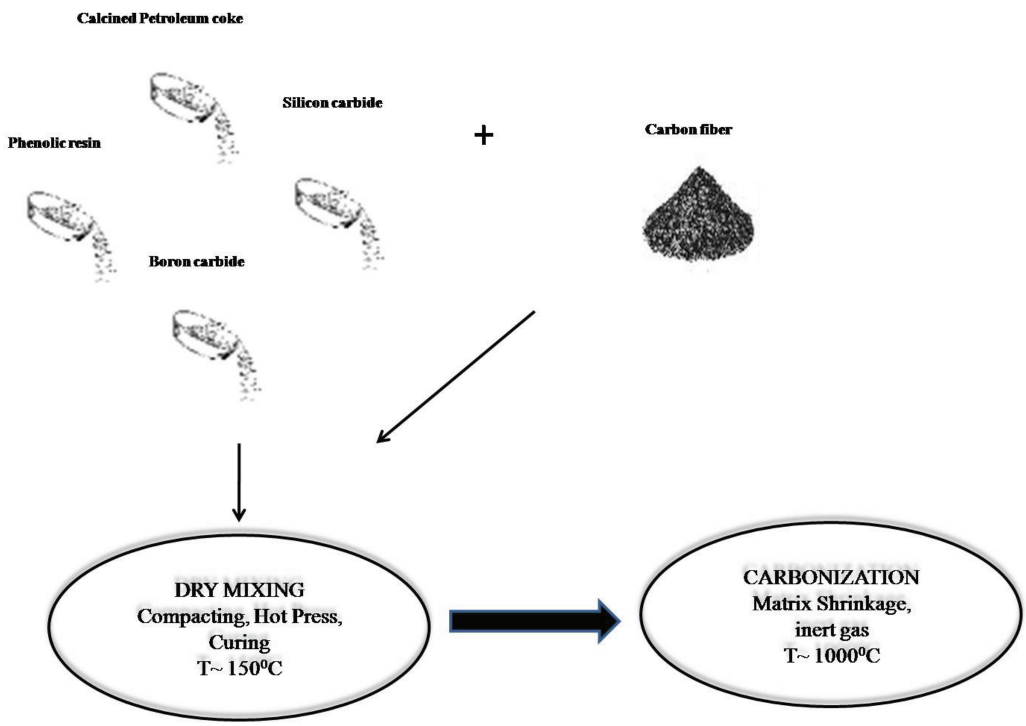

Fig 1. Schematic diagram for processing of ceramic reinforced phenolic resin based composite

obtained from Instron Bluehill software was used to calculate stress and strain. The tribological performance of each composite was evaluated by constant speed (an average linear sliding speed of 15.7×10^{-2} ms^{-1}). The schematic drawing of test rig for friction test is shown in Fig. 2. Different load condition, linear speed and static partner were varied with the content of carbon fiber. All the tests were performed at room temperature (without external heating to samples) in the atmosphere subject to a relative humidity. The friction force (and thus friction coefficient) was determined from the output of a strain gauge mounted on the arm carrying the pin. The temperature rises due to friction were measured using an Al – Cr thermocouple placed at a distance of 50 mm from one of the samples and 3 mm above the counter-face.

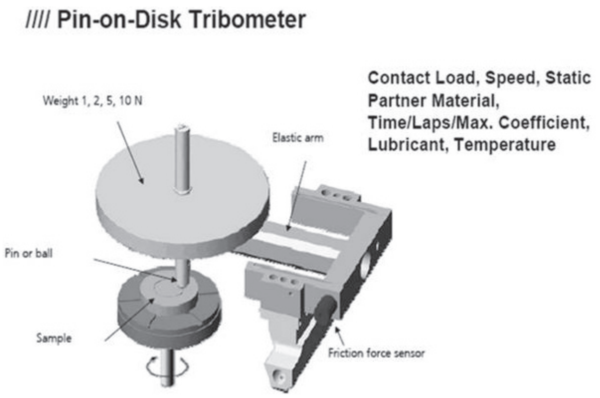

Fig. 2. A schematic diagram of Pin – on Disc - Tribometer

Results and Discussion

Effect of phenolic content on weight loss and dimensional changes

During carbonization of the composites, these are expected to lose weight and shrink due to pyrolysis of the phenolic resin. Change in thickness and weight of the different series of composites after carbonization are shown in Fig 3. As indicated in the figure, the amount of these changes increase with phenolic content. However, carbonization is primarily a process of pyrolysis of polymeric carbon precursor. The pyrolysis of polymers generally involves such processes as cleavage of C – H and C – C bonds to form reactive free radicals, molecular rearrangement, thermal polymerization, aromatic condensation and elimination of side chains with the evolution of gases as H_2O, CO, CO_2, H_2, etc [16, 17]. To minimize such adverse effects such as shrinkage, cracking and thermal stresses that may build up during carbonization, low carbonization rates (typically <15°C/min) are usually required. Below result is reasonable in view of the fact that the phenolic resin is the major constituent experiencing the carbonization process.

Effect of carbon fiber on physical property

Composites fabricated through hot pressing technique were subjected to bulk density and porosity measurement both before and after carbonization process. The results are seen in Fig 4. It is seen from fig. 4 that the introduction of carbon fiber (up to 10%) increases gradually the green density of the composites from 1.58 to 1.66 g.cm^{-3}. This increase in green density may be due to the presence of carbon fiber in the green mixture. The density increases after carbonization process as shrinkage is higher than corresponding weight loss during pyrolysis. Also, density increases with increase in carbon fiber content.

Effect of carbon fiber on the Mechanical properties

Mechanical properties of composites used in the present investigation are connected with their architecture, *i.e.*, particle size of the reinforcing agents, composition present in the composites, distribution and arrangement of reinforcements, characteristic length and distribution of fibers in the composite.

Fig. 5 shows the Rockwell hardness results performed on the surfaces of the fabricated ceramic reinforced phenolic resin based composites heat treated at 1000°C having different carbon fiber addition levels. It was found that heat treatment of green composites to 1000°C resulted in increased hardness. The increase in the temperature to 1000°C led to the bond formation between the fibers and the matrix which contributed to the increase in the hardness. It was confirmed by microscopic observation that the added CF adds to the bonding between the fibers and the carbon matrix.

During pyrolysis, the fiber/matrix interactions are so strong that the carbon matrix does not allow the carbon fibers to shrink away from it and it pulls the carbon fiber towards it which results in the formation of strong bonding between carbon fiber and the resin matrix. This bonding increases with the percentage of carbon fiber. The maximum compressive strength

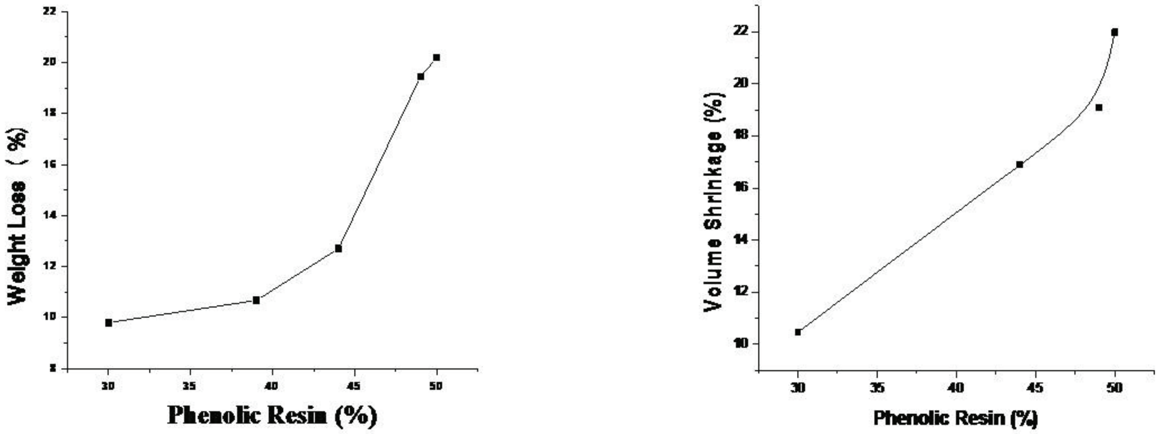

Fig. 3. Effect of content of phenolic resin of the ceramic reinforced phenolic resin based

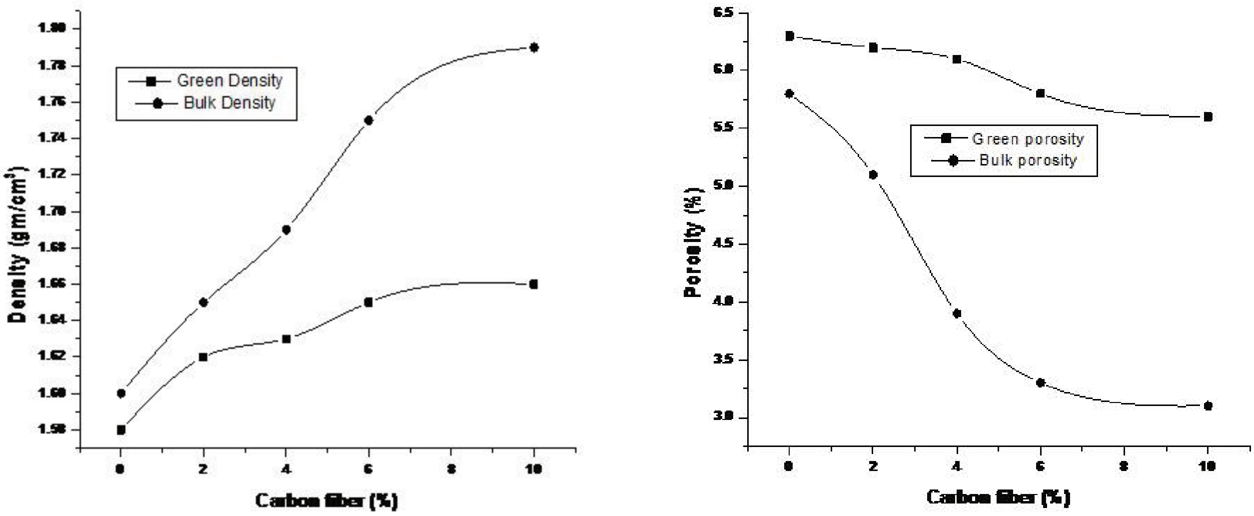

Fig. 4. Change in (a) Density and (b) Porosity of ceramic reinforced phenolic resin composites after and before heat treatment to 1000°C

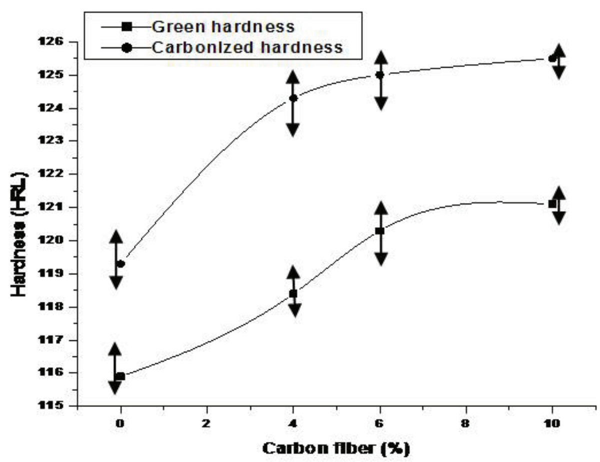

Fig. 5. Effect of carbon fiber on Rockwell hardness value heat treated at 1000°C

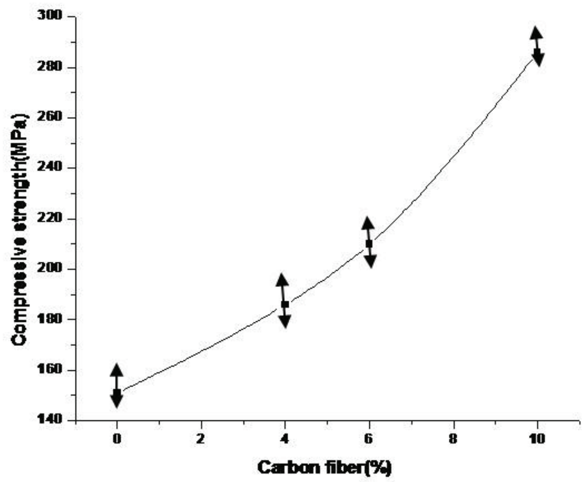

Fig. 6. Change in compressive strength with the percentage of carbon fiber present in the composites heat treated to 1000°C

of the ceramic reinforced phenolic resin based composites containing 10% carbon fiber was found to be 286 MPa as shown in Fig 6.

Tribological measurements

Tribological evaluations determines the friction coefficient from a combination of two materials known as substrate (disc form) and material identified as static partner (pin, ball, rod). The action results from the movement of a static partner on the flat substrate of corresponding studied material. In the pin-on-disc Tribometer the static partner in the form of a non rotating ball is fixed in the pin holder and is placed on a surface of the specimens. The pin is loaded with defined force at a defined distance from the centre of the sample. The tangential frictional force is caused by shear strength due to the asperities and their micro hardness. The axial tension loaded on the corpuscle arise the plastic deformation of both surfaces. In both cases of friction (adhesive and abrasive) the elimination of hills and transfer of material will depend on the ratio of materials and surface flatness of the composites. In case of dry friction, the test results usually depend on the following parameters: loading power, size of the contiguous surface, speed, temperature of the couple, environmental atmosphere, and, the mechanical and physical properties of the materials of the sample and the static partner.

Effect of sliding distance on the Coefficient of friction value of composites

Coefficient of friction (μ) varied significantly in the initial stage of testing, since the size of the contact area increased and the friction layer was developed on the surface. The variation of friction coefficient with test time, sliding distance and number of laps are given in figure 9. The characteristic value of μ evaluated from Pin-on disk Tribometer for different composites are shown in Fig 7. The curve showed initial drop before the 5th minutes of the test. This drop increases with the percentage of carbon fiber. After some interval of time the curves tend to follow steady-state behavior which can be conjectured that the morphological changes on the composites were completed and thus coefficient of friction value does not change much.

In case of higher load condition (4N), the coefficient of friction value shows an increasing behavior for composites without carbon fibers (NO). After 10th minute of the test, matrix material starts to get peel out from the reinforcing agents and the surface of the composite material having hard ceramic particles on the surface is exposed to the static partner which increases the coefficient of friction. Also, in case of N10CF initially there is low coefficient of friction which is due to the presence of higher amount of carbon fiber; then again after 12th minute of the test there is an increase in the coefficient of friction value which may be resulted

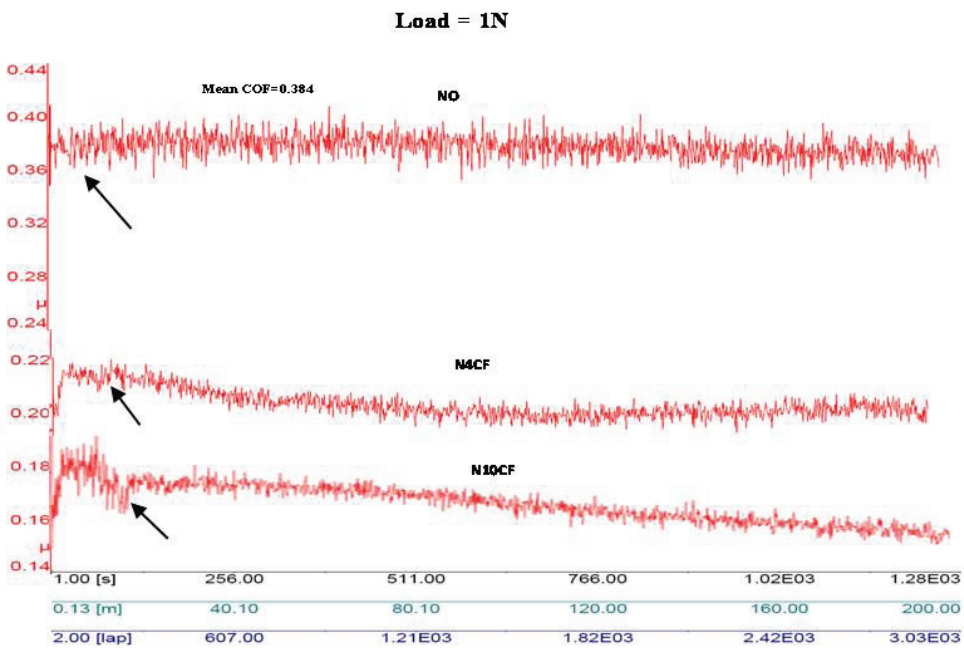

Fig. 7. Change of friction coefficient as a function of time, sliding distance and number of laps with 1N load condition

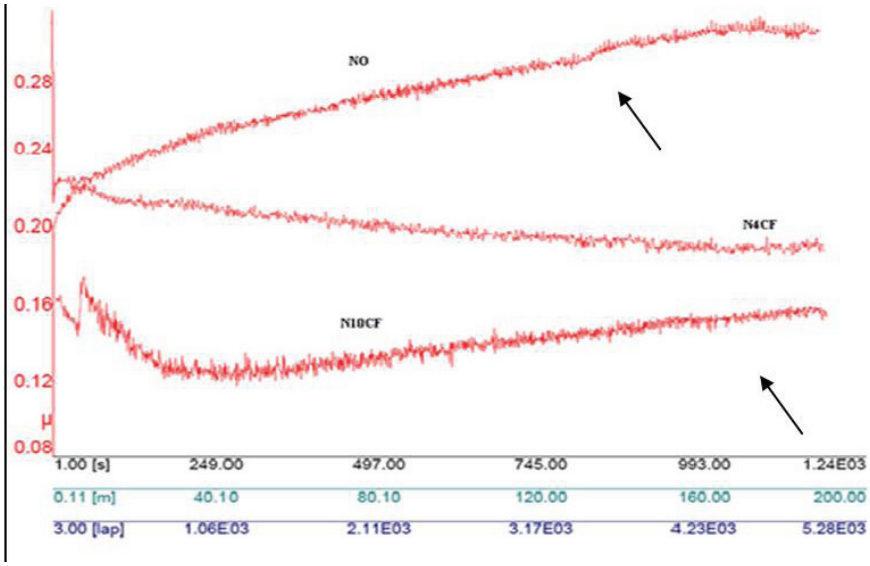

Fig. 8. Change of friction coefficient as a function of time, sliding distance and number of laps with 4N load condition

from contact between hard ceramic particles and static partner.

Effect of load on the coefficient of friction value

It is observed that the strength (Hardness and compressive) increased with the percentage of carbon fiber content as shown in Figure 5 & 6. Higher hardness is known to reduce a true contact area between the samples and the corresponding sliding material, thus contributing to lower friction. It is also observed that the carbon fiber addition decreased the friction coefficient of composites which are heat treated at 1000°C. The friction coefficient of composites containing 6 wt% of carbon fiber was measured to be 0.19. The lowest coefficient of 0.18 was observed at the carbon fiber content of 10 wt% heat treated at 1000°C. This was attributed to the multiple effects of the increased CF level and reduced true contact area. Finally, with increasing applied load the friction coefficient increases. In the present investigation, the overall composite performance controlled by the properties of the matrix and the interaction between the carbon fiber and the matrix.

Effect of linear speed on the coefficient of friction value

Increase in sliding velocity has invariably effect on the composites tribological properties which enhances the frictional force applied to the samples. It is interesting that higher sliding speed increases the coefficient of friction of composites. This increasing phenomenon hardly can be explained by the effect of the detachment of carbon fiber from the surface and contact of hard ceramic particles to the static partner. At higher linear speed, there is increase in the frictional force. At higher frictional force, carbon fibers get detached from the surface of the carbon matrix. As carbon fibers are short and hard in nature it is not easy for them to get stick on the surface of the composites. So, the coefficient of friction results due to the frictional force applied between static partner and ceramic constituents. Also, with increase in load condition the coefficient of friction value increases.

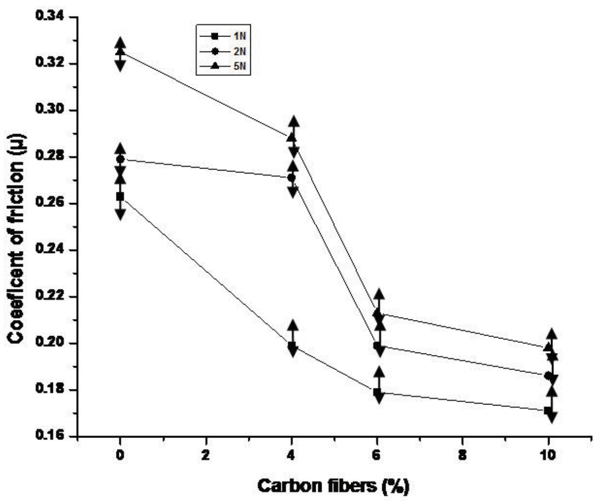

Fig. 9. Effect of load with the variation in the percentage of carbon fibers on the COF value of composites

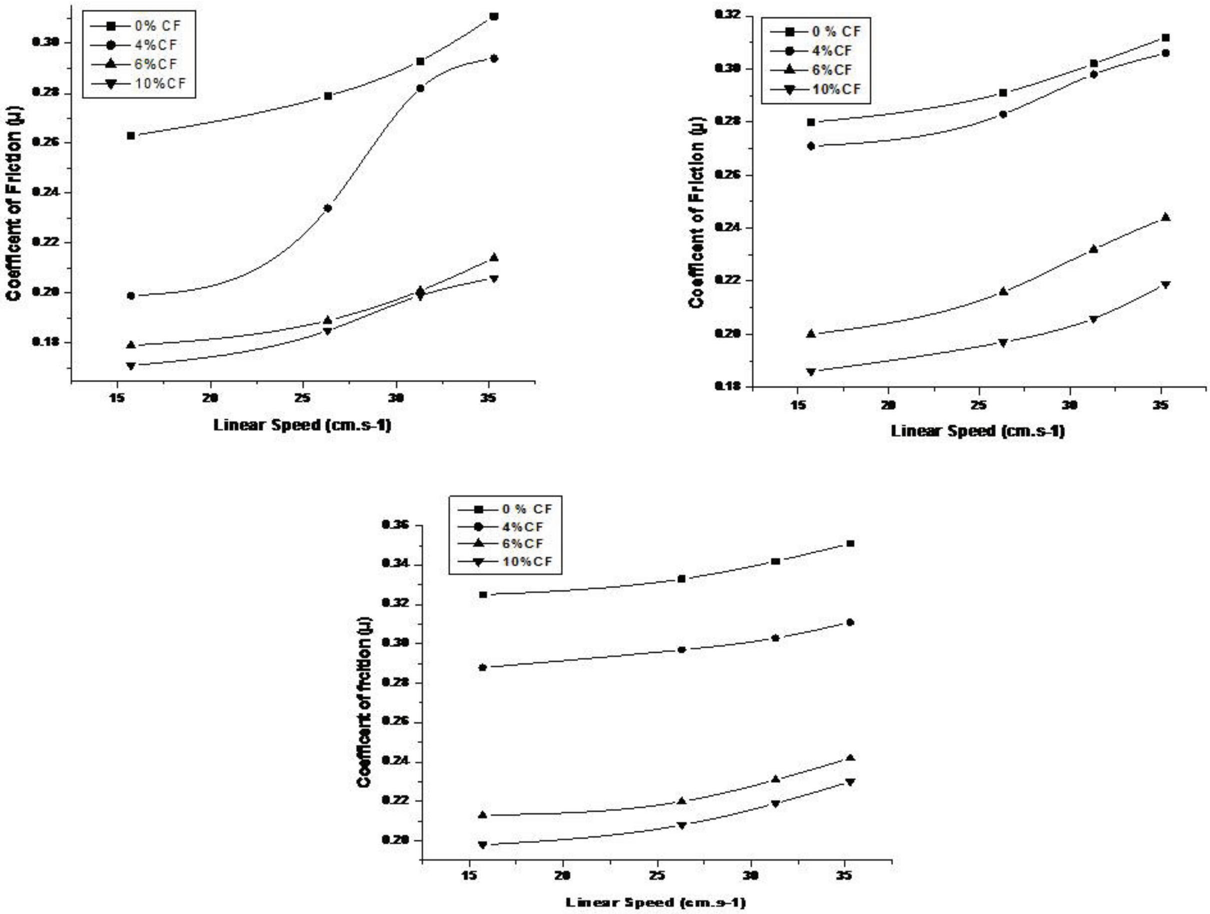

Fig. 10. Effect of linear speed with variation in the percentage of carbon at different load conditions

Conclusions

Ceramic composites have been processed through powder metallurgical route with phenolic resin derived carbon as matrix binder, ceramic particulates as filler materials and fiber as additional reinforcement. Effect of carbon fiber on the mechanical and tribological behaviors of the phenolic resin-based friction composites was investigated. Following conclusions are drawn:

(i) Dimensional shrinkage of the ceramic reinforced phenolic based composites are depend on percentage of phenolic resin. Higher the percentage of phenolic resin higher is the dimensional shrinkage;

(ii) Density of the ultimate composites was found to increase with the percentage of carbon fiber;

(iii) Mechanical strength of the composites tends to increase with carbon fiber. Composites having 10wt% of carbon fiber have maximum compressive strength;

(iv) Tribological evaluations shows that composites having 10 wt.% of carbon fiber has lowest coefficient of friction as compared to composites containing 2 wt% of carbon fiber. The increase in linear speed increases coefficient of friction.

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