Effect of antimony and SiC-I-Cu on mechanical and dry sliding wear pattern of phenol formaldehyde composites

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Abstract: Most of the brake linings used are based on metal fiber reinforced phenolic resin, which is a composite of 15-20 constituents and is generally called semi metallic brakes. In practice, asbestos, antimony, and their modified version of organic composite brake materials are used which possess good strength, resistance to temperature, chemicals and wear. In the present work, mechanical and dry sliding wear behavior of non-asbestos organic (NAO) friction composites namely; antimony and SiC-I-Cu filled phenol formaldehyde (PF) were studied following the ASTM standards. The tribological behavior was studied under dry sliding conditions using a pin-on-disc apparatus. Experimental results showed that there is a marginal improvement in the compression strength and hardness SiC-I-Cu filled phenol formaldehyde (SiC-I-Cu-PF) than that of antimony ones. Further, the coefficient of friction increased in SiC-I-Cu-PF composite. However, the k, increased with increase in sliding velocity and decreased with increase in the applied load. Scanning electron micrographs are used to analyze the fracture and worn surface morphologies.

Key words: Filler filled phenol formaldehyde, Mechanical properties, Coefficient of friction, Specific wear rate, Scanning electron microscopy.

1. Introduction
Non-asbestos organic (NAO) based friction materials are essentially multi ingredient systems (containing more than 10 ingredients, in general) in order to achieve the desired amalgam of performance properties [1]. Though the list of ingredients used for formulation of such composites exceeds the number 700 [2], these are classified into four major categories viz., binder, fibers, friction modifiers and fillers based on the major function they perform apart from controlling friction and wear performance. Binder is the heart of a system which binds the ingredients firmly so that they can perform the desired function in the friction materials. Fibers in combination are added mainly for strength, while friction modifiers are used to manipulate the desired range of friction. Fillers are of two types viz., functional fillers (to improve particular characteristic feature of composites such as resistance to fade, etc.) and space/inert fillers (mainly to cut the cost).

Phenolic resins (modified and unmodified) are invariably used as binder in friction materials due to low cost along with a good combination of mechanical properties such as high hardness, compressive strength, moderate thermal resistance, creep resistance and very good wetting capability with most of the ingredients. But these phenolics suffer from serious drawbacks and limitations such as evolution of noxious volatiles [3] (viz.NH3 (ammonia), HCHO (formaldehyde), etc.) during ambient temperature curing also, very short shelf life posing serious problem of storage and transportation, shrinkage, cracks and voids in the final products, need of catalyst which is mixed in the powdery resin before dispatching to the commercial market, etc. leading to increased cost. In spite of these facts, the choice of binders in non-asbestos friction materials is still largely limited to phenolics and its modified versions because of non-availability of right type of alternate resin along with viable cost.

Wear performance of friction materials depends on the inherent material properties such as chemical and physical nature of the ingredients and on the operating conditions such as pressure, speed, temperature, environment, counterface [4]etc. Many researchers have studied the friction and wear of composite brake materials. Wear of the materials and mean temperatures of the surfaces varied non-linearly as a function of load and speed [5]. Moreover, linear wear hypothesis was reported to offer a close approximation of the wear performance of friction materials and hence in the prediction of pad life [6]. Though it is well accepted that operating parameters and compositional changes in materials simultaneously influence the performance, no effort has been made to critically analyze the interactive influences of the individual parameters on the wear process of friction materials [7]. The present paper deal with the mechanical and wear behavior of multi-ingredient friction railway brake blocks, based on antimony and SiC, Fe and Cu fillers.
2. Experimental

2.1. Fabrication of the composites

The fabrication of composites containing fifteen ingredients was based on keeping parent composition of 12 ingredients (85%, w/w) constant and varying three ingredients, viz. silicon carbide, iron powder and copper powder (15%) in complementary manner. The parent composition contained binders- Linseed oil, Cashew nut oil, PF, Friction dust (30%), functional fillers- Fire clay, Plaster of pairs, Ferro silicon, (30%), frictional material - Rubber, Antimony, carbon(21%), lubricant – graphite (5%) and reinforcing material and catalyst (fiber glass, hexamine) (1%) each.

The required quantity of composites was obtained from the two main suppliers, Mysore pure chemicals and Maxin enterprise. These raw materials are prepared by subjecting it to pulverization process, material are feed in to hammer crusher machine, this machine brake up raw material through the collision of the fast rotating ram and materials.

The fabrication of composite brake block was manufactured by ‘Hot compression molding technique’. The process of manufacturing a composite brake block consists of series of operations including mixing, hydraulic pressing, cooling, post curing and finishing. The constituent ingredients, Fire clay, plaster of pairs, ferrosilicon, friction dust, rubber, antimony, carbon, graphite, hexamine and fiberglass were blend for 20 min in a mixer, to ensure the macroscopic homogeneity. The mixing sequence and time of mixing of each lot of ingredients lead to proper uniformity in the mixture. If mixing time is low, proper homogeneity cannot be achieved. If it is too high, it does not improve the homogeneity further. Hence, it has to be optimized which was done in the laboratory in earlier work. About 20 kg of the blend was discharged onto each performance, the composite are then separated to 1.5 kg of packets manually. The mold and die is initially pre heated and cleaned by thin layer of soap water (it acts as a barrier between the composite and the mould cavity). The back plates were coated with adhesive to secure bonding between the friction composite and plate, the perform pressure was varied between 5-10 ton for 5 seconds. The assembly was then subjected to hot pressing, which includes curing, at a pressure of 40 ton and temperature at 120°C for 40 min. After the prescribe time the composite brake. The pads were then removed and were then post-cured in an oven at 120°C for 17 h. The post curing operation is done to cure the residual resin. The surfaces of the pads were then polished with a grinding wheel to attain the desired thickness and smooth surface.

2.2 Hardness and Compression test

The hardness test is conducted on Shore D hardness testing machine as per ASTM D-2240 standards, the hardness test is done on antimony and ceramic fillers filled PF composite railway brake block material using diamond pyramid indenter at different points for ten trials each on the same surface and the hardness value is determined. A hydraulic operated crushing test was conducted to investigate the compressive force during the braking application, the test is conducted on hydraulic press testing machine as per RDSO standards on antimony and ceramic fillers filled PF composite railway brake block material for ten trials each on two specimens, the application of load was manual at constant rate till the fracture of block and the bonding strength of the composites are determined.

2.3 Dry sliding wear test

A pin-on-disc test apparatus was used to investigate the dry sliding wear characteristics of the composites as per ASTM G99-95 standards. The disc used is En-32 steel hardened to 62 HRC, 120mm track diameter and 8mm thick, with surface roughness of 10µm Ra. The tests were conducted by selecting test duration, load and velocity and performed in a track of 75 mm diameter. The test specimen are cut into required standard and placed parallel to the contact surface and to the sliding direction. The surface (8 mm X 8 mm) of the composites specimen makes contact to the counter surface. Prior to testing, the samples are rubbed over a 600 grade SiC paper to ensure proper contact with the counter surface. The surface of both the samples and disc are cleaned with a soft paper soaked in acetone before the test. The initial weight of specimen was measured in a single pan electronic weighing machine with least count of 0.0001g. The difference in the weight measured before and after test gives the sliding wear of composites.
specimen and then the volume loss was calculated. The wear test is carried out with variable sliding speed, load and sliding distance. The specific wear rate $K_s$ was calculated from the following equation.

$$ K_s = \frac{\Delta m}{\rho x L x D} \frac{m^3}{Nm} $$

where $\Delta m$ was the weight loss in g, $\rho$ the density in g/cm$^3$, $L$ the load in N and $D$ the sliding distance in m.

3. Results and discussion

3.1 Hardness (Shore-D)

Hardness test were conducted on the Antimony and SiC-I-Cu filled PF composite brake blocks using Shore-D hardness tester. Fig. 1 shows the measured surface hardness of the composite brake blocks. It is evident from the figure that hard ceramic fillers filled namely SiC-I-Cu reinforced PF composite had the highest hardness. It is attributed to the ceramic fillers having higher hardness which are compounded with the PF matrix.

3.2 Crushing strength

This test is conducted to determine the compressive force during the application of brakes, it is evident from the Fig. 2 crushing load of the S-I-Cu-PF composite exhibits better withstand to compressive force compared to Antm-PF composite.

3.3 Coefficient of friction

The friction material synthesized was subjected to a wear test by making use of a pin on disc wear tester. The coefficient of friction was found to vary for two loads namely 40 and 80 N. The coefficient of friction is governed by the relation;

$$ \mu = \frac{F}{N} $$

Where $\mu$ represents the coefficient of friction of the sample, $F$ the frictional force and $N$ represents the normal load. The tester was coupled to a computer to generate the results for the plot; the sample used for the test was of 8 mm diameter and is slid against a rotating steel disc. Initially a load of 40 N is applied on the sample against a speed of 236 rpm of the rotating disc. An initial period of 15 s is allowed for attaining stability. Beyond this, it is clear from the plot that the coefficient of friction is almost constant. A similar trend is observed for the samples at 80 N applied load. This clearly shows that the SiC-I-Cu-PF composite has exemplary resistance to wear under different loading conditions, and the coefficient of friction is high for SiC-I-Cu-PF composite compared to antimony filled PF composite. The prepared samples were found to exhibit excellent coefficient of friction as illustrated by the plot of coefficient of friction as a function of sliding distance in Fig. 3.
Fig. 3 Coefficient of friction as a function of sliding velocity of composite brake blocks at: (a) 40N and (b) 80N.

3.4 Wear volume loss

Plot of wear volume loss as a function of sliding velocity for antimony and SiC-I-Cu filled phenol formaldehyde (SiC-I-Cu-PF) composites for applied normal of 40 and 80 N are shown in Fig. 4 respectively. From the plot it was noticed that, the wear volume loss increases with increase in velocity for both composites. The wear volume loss of SiC-I-Cu-PF composites is lower than that exhibited by the antimony-PF composites under the condition of 3000 m sliding distance. The amounts of SiC, Fe and Cu in PF composite are 12, 1.5 and 1.5 wt % respectively. For this reason, the fillers loaded PF wear volume loss were small and wear volume loss had been caused mainly by matrix wear. Also, it should be noted that SiC-I-Cu-PF composite exhibited the highest wear resistance under different sliding velocity/loads. This behavior can be attributed to the presence of SiC, Cu and graphite particles, which act as effective barriers to prevent large-scale fragmentation of phenol formaldehyde (PF). This is also evident from the small size of the wear debris particles as determined by SEM analysis (Figs. 6 and 7). Several authors have discussed the role of SiC and as an effective filler material. The glass fiber and SiC strengthens the composite, while the graphite acts lubricant together providing enhanced wear resistance.

Fig. 4 Wear volume of Antimony and SiC-I-Cu filled PF composites at: (a) 40 N, 3000 m, and (b) 80 N, 3000 m.

The Railway brake blocks were made up of (a) fibrous materials, (b) ceramic particles, (c) anti-wear materials, (d) fillers and (e) binders, curing agents and heat and wear resistance additives. The matrix binder is a critical component. The limits of heat resistance and strength of the frictional material are governed largely by the heat resistance and strength of the matrix binder. PF matrix are highly brittle by nature, thus to reduce the brittle nature of the composite, specific ratios of epoxy resin and rubber particles are used. This improves the toughness of the resin-bonded mixture. The composite thus consists of PF and rubber that function as the base matrix. The matrix is strengthened by reinforcing materials of Fe short fibers and friction materials comprising of SiC. Graphite and cashew dust are added to act as anti-wear additives and the addition of ferro-silicon resins acts as a fire retardant. The rubber solution helps reducing the brittle nature of the phenol formaldehyde base. A catalyst agent in the form of hexamine is used to improve the curing time. Increasing the percentage of graphite content increased the wear resistance but at the cost of friction coefficient. The filler contents were chosen at percentages which were optimum for blending and at the same time provided the desired characteristics. Thus, the appropriate composition as indicated have been obtained after a number of iterations and trials and errors associated with them, thus optimizing the process requirements.
3.5 Specific wear rate

Fig. 5 (a) and (b) shows the dependency of the specific wear rate on different fillers present in PF polymer. It is evident that the presence of SiC-I-Cu in PF led to a remarkable decrease of specific wear rate. This kind of variation has been reported for glass fiber reinforced epoxy composites. The wear of antimony filled PF composite consists of two wear modes: polymer matrix wear, which includes matrix plastic deformation and cracks in the matrix and fiber wear, which involves fiber sliding wear, fiber cracking, fiber rupture, and fiber pulverizing. The reduction in specific wear rate with SiC-I-Cu in PF composite is due to the transfer film formed on the counterface. It is well known that the wear behavior of a polymer sliding against a metal is strongly influenced by its ability to form a transfer film on the counterface. During sliding, the transfer film decreases the adhesion between polymer composite and metal counterface, impairs the plowing action of metal asperities at counterface over the soft polymer surface. On the other hand, once the film is formed, subsequent interaction occurs between the SiC-Fe-Cu filled PF and a layer of similar material. As the phenol formaldehyde / SiC-Fe-Cu-graphite is a self-lubricant material so that the wear loss of SiC-Fe-Cu filled PF composite is closely related to the formation and development of a transfer film. At the start of sliding, all the asperities of the two surfaces are in contact with each other. As shear forces are applied, the asperities deform. The graphite particles protrude out from the surface of the sample. At first the Polymer matrix wears and only ceramic and glass fibers are in contact with the countersurface. As sliding velocity increases the wear rate decreases because at high load, the interface temperature increases causing the fiber to fracture and the resulting situation at the interface is complex owing to the fiber being trapped by the other ceramic and graphite fillers prevent as lubricant. The broken fibers wear the sample further due to third body abrasion. When polymer comes in contact, adhesive wear occurs and the wear rate increases. Further, the wear process is controlled by ceramic fillers reinforcement. Better wear resistance was obtained by the addition of SiC-I-Cu. During sliding, the graphite particles get smeared at the interface forming a graphite film which in turn reduces the specific wear rate. The temperature attained in high performance braking systems is hot enough to decompose PF and similar organics by high temperature oxidation. The PF first chars, which means it is converted to carbon accompanied by loss of mass, and then ablates into carbon dioxide.

3.6 Scanning electron microscopy

The scanning electron microscopic examination of worn surfaces of antimony and SiC-I-Cu filed PF composite samples against steel countersurface under 40 and 80 N loads and at 1 and 2.5 m/s sliding velocity are given in Figs. 6 a-d and 7 a-d. It is clear from figure 6 that there is a certain amount of void generation after the wear test. However, this void generation is localized and is minimal due to the excellent wear resistance exhibited by the composite (Fig. 6).

Fig. 6a–d presents the features of worn surfaces of antimony filed PF composites under different sliding conditions, i.e. 40 N, 1 and 2.5 m/s, 80 N, 1 and 2.5 m/s, respectively. At low load and lower sliding velocity, the worn surface was relatively smooth and associated with micro-cracks in the matrix (Fig. 6a). At higher sliding velocity, damage to the fiber and other hard fillers is higher resulting in more fiber removal from the surface (Fig. 6b). When the load...
increased up to 80 N, the wear loss and consequently the contact temperature were greatly increased, which caused an accelerative breakage of the matrix especially in the interfacial region. As a result, the surface damage remarkably increased with imprints left by the separating fibers (Fig. 6c and d). Accordingly, almost entire fiber/filler debris was observed on the countersurface. In this case, the fibers were removed with larger patches and underwent little wear. Moreover, the large fiber debris could further decrease the wear resistance of composite owing to a three-body abrasive wear effect. Therefore, the wear rate of the antimony filled PF composites was progressively enhanced at increased load and sliding velocity.

Fig. 6 SEM pictures of Antimony filled PF samples at (a) 1m/s, 40 N, (b) 2.5 m/s, 40 N,(c) 1m/s, 80 N,  and (d) 2.5 m/s, 80 N

Fig. 7 SEM pictures of SiC-Iron-Cu filled PF samples at (a) 1m/s, 40 N, (b) 2.5 m/s, 40 N,(c) 1m/s, 80 N,  and (d) 2.5 m/s, 80 N.
Fig. 7a–d shows the worn surfaces of SiC-I-Cu filled PF composites tested under 40 N, 1 and 2.5 m/s, 80 N, 1 and 2.5 m/s, respectively. In comparison with Fig. 6a–d, it was clear that the worn surfaces were much smoother at the same sliding conditions and the glass fiber detachment was greatly limited with the addition SiC, Fe, Cu and graphite particles. As shown in Fig. 7c and d, even under a relatively high load, the worn surface preformed relatively smooth and characterized by micro-surface damage are due to fatigue wear, which normally occurs at high temperatures and removes the surface layer by micro-cracks. Accordingly, the fiber debris resulted from composite (Fig. 7c) are much thinner and smaller than that of antimony filled PF under the same sliding condition. Therefore, with the additions of SiC, Fe, Cu and graphite particles, the fibers were maintained in the matrix with a very gradual wear process even at high load and velocity conditions and led to an enhanced load-carrying capacity of the composite. Also, Fig. 7d shows some kind of disintegration of the sample. Further, C-E filled with SiC, Fe, Cu and graphite shows more stable wear performance under all test conditions. It seems that although additional lubricants contribute to a stable development of the transfer film even at extreme sliding conditions, meaning that uniform transfer of graphite from the sample to the counterface is observed.

The samples (SiC, Fe, Cu and graphite filled PF) prepared was found to exhibit very high wear resistance, necessary for vehicle brake linings. It is also evident from the above results that the characteristics exhibited by the SiC, Fe, Cu and graphite filled PF composite has superior characteristics when compared to the antimony filled PF composite.

4. Conclusions

Based on experimental results the following conclusions can be drawn:

- The incorporation of the SiC, copper, graphite and iron particles in the polymer matrix as a secondary reinforcement increases the wear resistance of the composite brake block.
- Effect of variables i.e., load and sliding distance is more pronounced on the wear of the composite rather than sliding speed.
- It was also found to exhibit exemplary friction and anti-wear characteristics along with providing high temperature stability.
- The constituents used in the composite are extremely economical and are hence appropriate for industrial applications. The composite becomes even more economical when manufactured in bulk for industrial applications.

References


