

Effect of ceramic reinforcement particle on mechanical and tribological properties of sintered copper-based brake composite friction materials using pin-on-disc tribometer

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KEYWORDS	ABSTRACT
Brake friction material Copper Composite Friction TiC Wear	The main objective of this study is to investigate the role of titanium carbide (TiC_p) reinforcement particles in sintered copper-based brake friction material for medium-duty automotive applications. The different types of copper-based brake composite friction material containing 15, 20, 25, and 30 vol.% of TiC are prepared by conventional powder metallurgy technique. The experimental results show a change in the compositions of TiC _p has a significant effect on both mechanical and tribological properties of sintered copper-based brake friction material. Particularly, composites containing 25 vol.% of TiC _p have shown an increase in the friction coefficient of 21% and a reduction of almost 40% in wear as compared with the lowest TiC composition.

1.0 INTRODUCTION

In automobiles, the brake components play a major role in stopping the vehicle's motion under relative motion. Since the disc brake shows better performance in terms of heat energy dissipation than the drum brake, most vehicles considerably use disc brake as a major component in the brake system (Blau et al., 2003). Gray cast iron is used to make the rotor of disc braking systems because it has higher heat conductivity, a high damping capacity, and strong mechanical strength. Nevertheless, the required performance of the disc brake system namely safety and durability may not be achieved with a single brake lining material, under different operating conditions. To achieve sustainable friction and low wear at different working conditions, such as speed, contact load, temperature, and environmental situation, friction materials are essential in

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brake pad composition. Furthermore, to reduce the rotor's significant wear, the compatibility of the friction material with the rotor body is crucial. As brake linings, the brake pad typically comprises more than 10 different components. These ingredients frequently include abrasives, binder resin, fillers, property modifiers, reinforced fibers, and solid lubricants. The diverse types and relative amounts of these elements, which can be grasped by experience, empirical observation, or the experimental technique of trial and error, can be used to formulate brake pads. Among the various ingredients of brake lining materials, reinforced fibers, and abrasives are identified as major ingredients to achieve better performance of the brake system (Chan et al., 2004). However, fibrous ingredients are majorly responsible for improving the structural integrity and helping to maintain the friction level followed by a reduction in wear of the materials under various operating conditions. Zirconium silicate, alumina, silicon carbide, titanium carbide, boron carbide, quartz, iron oxides, multiwall carbon nanotubes, and other materials are frequently employed as reinforcement particles in friction material. Whereas solid lubricants like graphite, molybdenum disulfide, antimony trisulfide, copper sulfide, calcium fluoride, etc. are employed for commercial friction materials. Both (reinforcement particles and solid lubricants) protect the counter surfaces from excessive wear and reduce noise and vibration caused by stickslip action at the friction interface (Liew et al., 2008). As it supports at high temperatures and produces varied friction characteristics, choosing the right reinforcing particles for enhanced braking performance has been a crucial challenge for friction material developers. As a result, extra care has been taken in choosing the kinds and proportions of reinforcing particles.

Basavarajappa et al. (2006) investigated the dry sliding wear behavior of hybrid metal matrix composites under constant load and speed using a pin-on-disc experiment. Based on their investigation, they concluded that the composite's wear resistance is enhanced by the synergistic impact of several types of reinforcing particles. In addition, Riahi AR and Alpas AT (2001) observed that under the action of sliding, the wear of the aluminum matrix composite has considerably changed from severe to mild with the development of a new tribolayer over the contact surface. Biswas et al. (1981) revealed that the wear properties of unreinforced Al-Si composites showed better performance than those of composites with 2.7-5.7% graphite. Rohatgi et al. (1997) reported the influence of SiC reinforcement particles as well as the generation of graphitic layers on the interacting surface during sliding support to improve the bulk mechanical and tribological properties of Al-10SiC-6Gr composites. Hazim Sharudin et al. (2019) concluded that a higher percentage of copper among the combinations in brake friction material supports enriching the tribological properties in terms of friction and wear over commercial carbon brake friction material. Tjong SC and Lau KC (1999) studied the properties and wear of TiB₂/Al-4%Cu composite by hot isostatic pressing. They concluded that the amount of material removal from the composite has significantly reduced with the addition of reinforcement particles. Raja P and Ramkumar P (2018) studied the tribological behaviour of Cu/SiC/Gr hybrid composite brake friction material by changing the compositions of multiwall carbon nanotube (MWCNT) for medium-duty automotive applications using pin-on-disc experiment. They have observed that the addition of MWCNT has a significant improvement in the friction coefficient and wear behaviour of hybrid composite brake friction material. This was attributed to the increase in wear resistance of the hybrid composite material. Singh et al. (2021) studied the effect of friction modifiers' compositions (such as talc and graphite) on the tribological characteristics of the Cu-Sn alloy/Al₂O₃ brake material. They have reported that the incorporation of friction modifiers shows better mechanical and tribological properties of the composite material. However, higher wear resistance of Cu-Sn alloy/Al₂O₃/15 vol. % Gr was achieved due to the formation of thick

lubricating film as a protective layer to the contact surface than Cu-Sn alloy/Al₂O₃/10 vol. % Gr and 2.5 vol. % talcum powder and Cu-Sn alloy/ $Al_2O_3/10$ vol. % Gr and 5 vol. % talcum powder. P. Zhang et al. (2020) studied the braking performance of copper-based brake pads with the range of 0-1.2 wt. % of carbon fiber (CF) using a reduced-scale dynamometer. They have reported that 0.4 wt. % of CF has shown better wear resistance and stable coefficient of friction during braking as a result of high plastic deformation resistance developed on the tribo-film surface. Nalin Somani and Nitin Kumar Gupta (2022) observed that the addition of TiC nanoparticles helps in improving the mechanical and tribological properties of a copper-based composite. It also enriches the composite's wear resistance by forming oxide layers and mechanically mixed layers as a protective element to the surface. Numerous studies have been conducted on various types of hybrid composite especially on Fe matrix and Al matrix for various applications such as grinding tool machinery, automotive, and aerospace industry. Many researchers have reported that the incorporation of ceramic reinforcement particles like SiC, TiC, and B₄C, etc. into the matrix material has a significant level of improvement in wear resistance, strength, and compatibility between the reinforcement particles and matrix. Some recent studies have focused on improving the material characterization and performance of friction and wear behaviour of Fe/SiC/Gr, Al/SiC/Gr, Cu/SiC/Gr, Cu/Al₂O₃/Gr, and Cu/TiC/Gr by changing the percentage of soft and hard reinforcements in the matrix for limited applications (Jaswinder Singh, 2016; Mohammad et al., 2011; Rajkumar et al., 2011). To the best of our knowledge, the combination of the sintered copper-based matrix with TiC reinforcement particulate has not been developed and studied for automotive applications. Therefore, the current study intends to examine the tribological impact of TiC reinforcement compositions on sintered copper-based brake friction material using a pinon-disc tribometer for medium-duty automotive applications.

2.0 EXPERIMENTAL METHODOLOGY

2.1 Materials Selection

Materials used for preparing brake composite friction material were purchased in the form of powder from Shriram Enterprises Private Limited, Chennai, Tamilnadu, India. These powders namely copper, titanium carbide, graphite, and barium sulfate were obtained with purity & particle size of 99.50 % & ~85 μ m, 99.50 % & ~2 μ m, 99 % & ~46 μ m and 98 % & ~45 μ m respectively and morphology structure of TiC particle is also shown in Figure 1 using field emission scanning electron microscope (FESEM). Cu/TiC/Gr hybrid brake composite friction materials are prepared using 49 vol.% copper (Cu) as a matrix, 7.5 vol.% graphite (Gr) as a solid lubricant, 1.0 vol.% Grey tin (Sn) as an additive along with varying volume ratios of 15%, 20%, 25% and 30% titanium carbide (TiC) as reinforcement; the remainder is barium sulfate (BaSO₄) as filler, which is represented in Table 1.



Figure 1: Micrograph image of TiC ceramic particles using FESEM.

Table 1: Different types of brake composite friction materials (Note: All values are in volume percentage (vol. %)).

Base Compositions: Cu =49; Gr = 7.5; Sn=1					
Matariala	Types				
Materials	Α	В	С	D	
TiC	15	20	25	30	
BaSO ₄	27.5	22.5	17.5	12.5	

2.2 Synthesis of Cu/TiC/Gr Hybrid Brake Composite Friction Material

The conventional powder metallurgy technique is used to prepare the brake composite mixtures (Cu/TiC/Gr) by adopting the following steps in sequential order: dry mixing, blending, compaction, and sintering. Finally, the prepared samples have undergone testing to characterize the microstructural, mechanical, and tribological performances of the composite.

2.2.1 Ball Milling

All the constituents in the brake composite material are manually mixed by their relative amounts and are then transferred into a planetary ball mill. For this, tungsten carbide balls with powder to ball ratio of 1:10 are employed. Ball milling is done at a speed of 300 rpm for 2 hours duration. Ball milling grinds and blends the components into much smaller particles ensuring that they are properly mixed and homogeneous.

2.2.2 Compaction

The compaction process compresses the fine particles to a solid shape and improves the density resulting in imparting mechanical strength of the sample. A single die uniaxial hydraulic press (10 tons) is used for this process. The applied load is 6 tons (120 bar) at a ramping speed of 4 mm/sec. Finally, the dimension of the green compacted sample is in diameter and height of 8 mm and 10 mm respectively.

2.2.3 Sintering

It makes the sample into a solid mass by heating. It fuses different material particles and forms a solid piece with enhanced strength. Sintering is done in a hot tubular furnace at 1400°C with the support of an argon atmosphere. In order to achieve better mechanical and physical properties,

the effect of sintering on copper-based brake composite samples has been studied and optimized (Raja, P et al., 2018). Hence, the samples were treated at a sintering temperature of 1000 °C for 3 hours duration, with a heating proportion of 5 °C/min and a cooling proportion of 3 °C/min. Figure 2 shows the preparation stages of the Cu/TiC/Gr hybrid brake composite sample.



Figure 2: (a) Ball milled; (b) Sintered sample; (c) Cross-section and Sample dimension of Cu/TiC/Gr hybrid brake composite sample.

2.3 Material Characterization and Testing

The bulk density of the composites was evaluated using the Archimedes principle following the procedures of compaction and sintering. The void fraction was determined by dividing the difference between the theoretical density, estimated using the law of mixtures, and the actual density by the theoretical density. A minimum of three samples were used in the density measurement for repeatability. Using Micro Hardness Tester-Wilson equipment (Wolpert Group-German) and the ASTM E92 standard, the hardness of the composites was determined. The composite samples were tested for hardness at various points using a square base, pyramidshaped diamond indenter with an angle between opposing sides of $=136^{\circ}$ and a dwell time of 10 seconds. The average of five indentation values recorded at various points on the specimen is used to determine the micro-harness of the composites. According to ASTM E9 standard, a compression test is performed in a universal testing machine (SHIMADZU-AG-50KNXD Plus, Japan). The Materials Science Research Centre (MSRC), IIT Madras, has an optical microscope (OM) and a field emission scanning electron microscopy (FESEM) capability that was used to investigate the distribution of reinforcements in the Cu matrix and the morphology of composite samples. The composite samples were put through testing and their tribological properties were evaluated during dry sliding using the pin-on-disc (PoD) tribometer rig (Ducom Instrument, Bangalore, India). The wear test was conducted using a load and speed of 5 m/s and 50 N, respectively, for medium-duty commercial vehicle applications. For wear testing, the ASTM G99-05 standard is used. The composite pin samples have the following measurements: $8mm \times 8mm$ × 10mm, respectively. A grey cast iron disc (Grade: SAE J431, C: 3.35-3.60%, Si: 1.6-2.3%) of 165 mm diameter and 8 mm thickness is used as a counter body (rotor) in the PoD tribometer rig. The disc's surface roughness ranged from 0.5 to 1.0 µm, while its hardness ranged from 187 to 241 BHN.

Each test was conducted at the atmospheric temperature and an applied normal load of 50 N (an initial load of 10 N with increments of 10 N) and a constant speed of 5 m/s (Mohammad et al., 2011 & Chaurasiya et al., 2023). The total duration of the study is 40 minutes in which the first 10

minutes is considered for the ramping of the load known as the running-in period; the remaining as a steady-state period. All tests were repeated thrice for repeatability. Both the surfaces (pin and disc) were cleaned with acetone after each test to get rid of the debris, and they were then dried. To calculate the amount of wear lost for each test in tribological investigations, the cylindrical pins were weighed before and after the tests using a high-resolution electronic scale (Citizen CX 165 Analytical Lab Balance) with an accuracy of 0.1mg. The coefficient of friction (μ) of composite samples was determined from the applied normal load (N) and the observed tangential frictional force (F), and an average of three values with an error bar was plotted for a steady-state period of 30 minutes. The schematic representation of sample synthesis by powder metallurgy process and the experimental procedure is shown in Figure 3.



Figure 3: Schematic diagram of the experimental process.

3.0 RESULTS AND DISCUSSION

The performance of brake composite friction materials made using a conventional powder metallurgy process and reinforced with various volume percentages of titanium carbide is discussed in this study. To better understand the behavior of sintered copper-based brake composite materials, a thorough examination of the microstructural, physical, mechanical, and tribological (friction and wear) properties was conducted.

3.1 Microstructural Analysis

Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive Spectroscopy (EDS) analyses were used to examine the microstructure of sintered copper-based brake friction composite samples in both their developed and worn-out states. Moreover, composite samples were subjected to X-ray Elemental Mapping (EM) to better understand the distribution of the various ingredients found in the composite. As depicted in Figure 4, FESEM micrograph pictures of the four distinct titanium carbide compositions in the Cu/TiC/Gr hybrid brake composite friction material were acquired for all the samples.



Figure 4: FESEM micrograph of developed Cu/TiC/Gr composite samples: (a) Type A; (b) Type B; (c) Type C and (d) Type D.

It is noted from developed samples that Copper, TiC, and BaSO₄ particles are identified in the micrograph. FESEM micrograph also reveals the flake-like structured TiC particles and white with cubic structured BaSO₄ particles are present in the copper matrix. As an increase in the volume percentage of TiC particles, it is observed the densely packed arrangement between the reinforcement particles and copper matrix, hence this arrangement most likely shows a good interfacial bonding between them. Initially, at 15 and 20 vol.% TiC compositions, the presence of voids is higher in the matrix. It is mainly due to the larger size (~45 μ m) and relatively higher volume percentage of BaSO₄ particles compared with the smaller size (~2 μ m) and lesser volume percentage of TiC particles that induce more porosity at the interface. While, at higher

concentrations of TiC, lesser voids are seen, which probably indicates that there is good compatibility between TiC and $BaSO_4$ reinforcement particles with the existence of graphite in the copper matrix. Hence, it is crucial to understand that as TiC's volume % rises, the interparticle distance between TiC and $BaSO_4$ decreases, resulting in a uniform distribution of hard reinforcement TiC particles on the copper-based matrix.

3.2 Elemental Mapping Analysis

X-ray elemental mapping analysis was carried out to identify whether all particles in sintered brake composite samples are homogeneously distributed in the copper matrix. Figure 5 shows four different types of elemental (color) mapping of developed Cu/TiC/Gr composite samples with varying volume percentages of titanium carbide as primary reinforcement particles. Figure 5(a & b) represents the elemental mapping of type A of 15 vol.% TiC and type B of 20 vol.% TiC in copper-based brake composite samples respectively, where thick leaf green indicates copper matrix region, indigo blue, light blue, and violet colors regions as primary reinforcement elements of titanium, barium and graphite particles respectively. Further, elemental mapping of Type C of 25 vol.% TiC and Type D of 30 vol.% TiC is shown in Figure 5(c & d) respectively. It can be seen from Figure 5(c & d) that the light green region is represented as a copper matrix whereas sky blue, pink, and dark blue indicates titanium, barium, and graphite as primary and secondary reinforcement region respectively. From these figures, it is confirmed the homogeneous distribution of the reinforcement particles with copper matrix in all four different compositions. Similar to the microstructure analysis, a decrease in the matrix's porosity is seen when the volume percentage of TiC increases.

3.3 Physical and Mechanical Study

Table 2 displays the variation of densification, micro-Vickers hardness, and compressive strength with a varying volume percentage of titanium carbide in Cu/TiC/Gr hybrid brake composite friction material. It is clearly seen from Table 2 that except for type D, there is a significant rise in both physical and mechanical properties of Cu/TiC/Gr brake composite with an increase in TiC (vol.%) reinforcement particles to the copper base matrix material. In the case of 25 vol.% TiC, higher hardness was achieved and it might be due to the homogeneous distribution of particles and also the effect of better bonding achieved between reinforcement particles and copper matrix. Further, it should be understood from the microstructural analysis that at 25 vol.% TiC reinforced composite (Figure 4c), the TiC particles are uniformly distributed in copper base matrix material while at 30 vol.% TiC reinforcements (Figure 4d), partial agglomeration of particles in some regions as well as weak bonding to the matrix which influences the possibility of the indentation falls on the pores and hence the hardness of 30 vol.% TiC composite is reduced. Therefore, it is significant to note that the addition of hard reinforcement titanium carbide particles has a strong influence on the mechanical and physical properties of the composites. Among the combinations, Type C, which is 25 vol.% TiC in the Cu/TiC/Gr composite shows better physical and mechanical properties in sintered copper-based brake composite friction materials.



Figure 5: Elemental mapping of developed Cu/TiC/Gr composite samples: (a) Type A; (b) Type B; (c) Type C and (d) Type D.

Types	Densification (%)	Micro-Vickers Hardness (VHN)	Compression Strength (MPa)
А	60	21	20
В	63	24	22
С	67	29	25
D	65	25	23

3.4 Friction and Wear Analysis of Cu/TiC/Gr

When the volume percent of TiC increases, the mean coefficient of friction gradually rises, as can be seen in Figure 6. Also, when the TiC composition of the Cu-based brake composite samples is increased from 25 vol.% to 30 vol.%, there is no distinct increase in the coefficient of friction. Under the high load and sliding speed condition, harder TiC-reinforced particles from the copper matrix act as a load-bearing element but are abrasive between the contact surfaces. Further, the

protruded effect of TiC reinforcement particles rubbing against the cast-iron disc leads to a rise in friction with an increase in the volume percentage of TiC on the composites (Somani et al., 2021). However, the samples C and D types show almost the same higher coefficient of friction of 0.5542 and 0.5560 respectively among the combinations. A similar effect is also observed in hardreinforced SiC particles on aluminum-based matrix composite (Basavarajappa et al., 2006). These frictional values are satisfied more than the desired requirements of the coefficient of friction (0.3-0.45) for commercial automobile applications (Mohammad et al., 2011).



Figure 6: Mean Coefficient of friction of Cu/TiC/Gr brake composite samples with a varying volume percentage of TiC.

With a constant load of 50 N and a sliding speed of 5 m/s, Figure 7 shows the analysis of the effect of the volume % of TiC reinforcement particles on the wear behavior of Cu/TiC/Gr brake composite samples. It should be observed that when the volume % of TiC particles in the copper matrix has increased, the wear loss has decreased dramatically. It is also observed that the Type C (25 vol.% TiC) sample shows a relatively lesser wear loss of ~0.233 grams than the other three combinations. It is mainly attributed to the effect of improvement in hardness up to 25 vol.% TiC in the copper matrix; consequently, the wear resistance of Cu/TiC/Gr hybrid composite has increased. Moreover, the effect of an increase in the hardness of the composite helps in the reduction of localized plastic deformation at the contact surface during the sliding process. It is also noted from Figures 4(c) & 5(c) that the effective distribution of reinforcement particles in the Cu matrix contributes a major role in showing less wear loss in the Type C combination. In addition, the protrusion of secondary hard-phase particles can influence the formation of a protective layer on the contact surface. Hence, the incorporation of hard TiC reinforcement particles, which depict the act as load-bearing elements to the composite during sliding, controls the loss of material over the surface.



Figure 7: Wear loss (gram) of Cu/TiC/Gr brake composite samples with a varying volume percentage of TiC.

3.5 Post-Test Analysis

Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-ray Spectroscopy (EDX), and Elemental Mapping (EM) are employed to analyze the worn-out surface of Cu/TiC/Gr brake composite friction materials. Having analyzed the wear characteristics, the potential wear mechanisms of composite pin samples with a varying volume percentage of (15-30 vol.%) TiC under conditions of a constant load and sliding speed

3.5.1 Worn Surface Analysis of Type A Composite

The typical surface shape of worn Cu/15 vol.% TiC/Gr hybrid brake composite material is shown in Figure 8. The worn surface clearly showed a continuous plastically deformed layer was created on the sample surface due to the reduced TiC content. Maximum stress is produced in the direction of sliding with a strong load of 50N, which may be shared by fewer TiC particles in the matrix. As seen in Figure 8(c), additional ferrous oxide layers developed from the disc surface and adhered to the composite pin surface during sliding. As a result, it is confirmed that the primary mechanisms for the Cu/15 vol.% TiC/Gr composite sample include plastic deformation.



Figure 8: (a) FESEM micrograph; (b) EDX spectrum and (c) EM of worn surfaces of Type A composite.

3.5.2 Worn Surface Analysis of Type B Composite

Figure 9 depicts a FESEM micrograph of a Cu/20 vol.% TiC/Gr hybrid composite material's worn surface. With the increase in the volume percentage of TiC, it exhibits a plastically deformed surface with the production of microcracks in the composite pin surface. Moreover, an oxide layer exists on the contact surface, as shown by elemental mapping analysis of worn-out samples. When the volume percentage of hard reinforcement particles is increased to 20% in a TiC composite, the morphology of the worn surfaces gradually shifts from a plastic deformation to a delamination wear mechanism due to the higher stress caused by the maximum load along the sliding direction in the formed oxide layer.

3.5.3 Worn surface Analysis of Type C Composite

Figure 10 displays the worn micrograph of Cu/25 vol.% TiC/Gr. The sample surface is found to generate a tougher TiC layer with less oxide at a 25 vol.% TiC concentration, protecting the pin surface from plastic deformation and exhibiting reduced wear among the compositions, which is further supported by an elemental mapping image as shown in Figure 10(c). It should be highlighted that plastic deformation decreases as the volume percentage of hard TiC reinforcement rises owing to the increased load-bearing effect of pulled-out TiC elements. Moreover, the oxide layer formation on the pin surface was decreased by up to 25 vol.%.

3.5.4 Worn Surface Analysis of Type D Composite

The worn micrograph examination of the hybrid Cu/30 vol.% TiC/Gr brake composite material is shown in Figure 11. The morphology of worn-out surfaces changed with an increase in hard TiC reinforcement, as is evident from FESEM photos. The loose, fractured oxide particles

in the direction of sliding show that thicker delamination has been seen on the pin surface. As seen in Figure 11(c), these loose particles are a mixture of oxide debris from a counterpart with pulled-out TiC from the pin. The micrograph's thicker and more compacted layer supports delamination as the primary wear mechanism in 30% of the TiC composite.

Hence, it has been found that of the four compositions of TiC (vol.%), the 25 vol.% TiC composite has the maximum wear resistance. Therefore, based on the above-given reasons related to the wear mechanisms study, Type C (Cu/25 vol.% TiC/Gr) composition is chosen as optimum among the other three combinations of copper-based brake composite friction materials.



Figure 9: (a) FESEM micrograph; (b) EDX spectrum and (c) EM of worn surfaces of Type B composite.



Figure 10: (a) FESEM micrograph; (b) EDX spectrum and (c) EM of worn surfaces of Type C composite.



Figure 11: (a) FESEM micrograph; (b) EDX spectrum and (c) EM of worn surfaces of Type D composite.

CONCLUSIONS

From the analysis of the work, the following important points have been concluded:

- a. Based on formulation, Cu/TiC/Gr brake composite friction material has been successfully fabricated by the conventional powder metallurgy process.
- b. The physical and mechanical qualities notably rise when the copper matrix's volume percent of TiC_p reinforcement increases. The inclusion of hard ceramic reinforcement particles (TiC) also leads to an improvement in the bonding between the matrix (Cu) and the reinforcement (TiC_p), which increases the strength and hardness of the copper-based hybrid brake composite friction material by up to 25 vol.% of TiC_p.
- c. The dry sliding wear test for constant load and speed revealed that 25 vol.% of TiC_p reinforcement compositions exhibit an increase in friction coefficient of 21% and reduction of wear of about 40% as compared to the lowest TiC composition in Cu/TiC/Gr brake composite friction material-
- d. Based on the mechanical and tribological analysis, Type C of Cu/25 vol.% TiC/Gr shows an optimum volume percentage of TiC in sintered copper-based brake friction material for medium-duty automotive applications.
- e. From the microstructural analysis, delamination mechanisms are found to be predominant on the worn-out surface of the composite samples.

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