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Technical Report

Synergistic effect of tungsten disulfide and cenosphere combination on braking performance of composite friction materials

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ABSTRACT

Tungsten disulfide (WS_2/TDS) based cenosphere (Cn) filled friction composites with varying cenosphere to WS_2 ratio (Cn/TDS) were fabricated by compression molding of phenolic resin based dry formulation mix and evaluated for their thermal, thermo-mechanical and tribological performances. The loss and revival of braking friction effectiveness due to heating or cooling of the disc termed as fade and recovery performance have been characterized on a Krauss friction testing machine following ECE R-90 industrial standards. The fade performance remained dependent on Cn/TDS, where enhanced fading could be correlated to lower Cn/TDS value accompanied with broader frictional fluctuations i.e. μ_{max}–μ_{min}. A decrease in the frictional-recovery response ensued with increase in Cn/TDS. Dynamic mechanical analysis revealed an increase in storage modulus till 2.5 wt.% of TDS loading followed by consistent decrease whereas two distinct peaks in loss modulus plots that are composition independent have been observed. Scanning electron microscopy revealed the worn surface morphology associated with the dynamics of contact patches formation and deformation vis-a-vis friction layer formation as integrally responsible for the observed friction performance. Energy dispersive analysis of X-rays (EDX) enabled compositional analysis of the friction layer viz. Fe, W, Si, and Al content which may have a mechanistic role in controlling phenomena like, disc rubbing, lubricity, porosity, and hardness of friction layer formed during braking application.

1. Introduction

The reliable braking effectiveness of friction composites at elevated temperatures is crucial as far as the heavy vehicle dynamics is concerned under extreme braking load and speed conditions. Typically the friction requirements of heavy commercial vehicles (HCV) and light commercial vehicles (LCV) lie in the range of ~0.30 to 0.35 depending on the size of the vehicle and total road-tyre contact situations. Therefore, the incorporation of graphitic solid lubricants in friction formulations up to a reasonable level of loading is employed [1]. However, the lower lubrication effectiveness at elevated temperatures of graphite limits its potential as a high temperature lubricant. In order to avoid this problem, metal dichalcogenides/monochalcogenides like molybdenum disulfide and lead sulfide are used as potential lubricant alternatives to graphite though their usage is temperature-restricted [2]. Gudmand-Hoyer et al. [3] while investigating on performance of automotive disc brakes with solid lubricants on dynamometer simulating real life car braking situations have revealed that addition of Cu_2S caused gradual loss of friction while PbS and Sb_2S_3 stabilised the friction irrespective of the temperature. In the absence of metal sulfides as solid lubricant the friction level invariably tended to increase with the temperature. On the similar lines Cho et al. [1] have reported the role of solid lubricants such as graphites, Sb_2S_3 and MoS_2 in a combinatorial manner on the performance of organic friction materials. It was concluded from those studies that Sb_2S_3 and graphite combination improved friction stability and fade resistance whereas Sb_2S_3 and MoS_2 combination proved detrimental from wear resistance fade resistance and resistance to disc thickness variation (DTV) point of view. The DTV was ascribed to sulfuric acid assisted corrosion which may have been produced by the oxidation of MoS_2 in presence of water molecules in air. On the other hand, the presence of Sb_2S_3 may cause high DTV due to formation of the oxidized product of Sb_2S_3. Pronounced fade resistance have been reported in friction material with Sb_2S_3 and MoS_2 in contrast to those with graphite at elevated temperatures. Kim et al. [4] while assessing on complementary effect of solid lubricants in potassium titanate whiskers reinforced non-asbestos organic (NAO) brake lining materials with various combinations of graphite and Sb_2S_3 have showed that high concentration of graphite to be causing better fade resistance and friction stability during high temperature friction test. The use of chalcogenide compounds such as Sb_2S_3, ZnS, PbS and Cu_2S to mitigate squeal noise and braking induced vibrations was reported by Jacobson and co-workers [5]. Lee et al. [6] have reiterated the effect of Sb_2S_3 in improving

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Nomenclature

μ_P average of coefficient of friction in cold, fade and recovery cycle
μ_R highest coefficient of friction in the recovery cycle i.e. $\mu_{R/}\mu_P \times 100$
μ_F minimum coefficient of friction in fade cycle after 270 °C i.e. $(\mu_P - \mu_F)/\mu_P \times 100$
$\mu_{max} - \mu_{min}$ friction fluctuation ($\Delta\mu$) as the difference between the maximum and minimum friction in the three cycles (cold, fade and recovery)

the fade resistance by prompting the formation of smooth transfer film on disc surface at elevated temperatures, apart from exhibiting lower stick–slip propensity than the material with graphite. Jang and Kim et al. [7] have reported that the use of Sb_2S_3 in combination with $ZrSiO_4$ may enhance friction stability with some extent of torque fluctuation during braking especially when local micro-structural inhomogeneities due to removable friction-film debris are present.

Theoretically, the use of metal sulfides or metal dichalcogenides has been reported to facilitate structural transformation in terms of changing MoS_2 more readily so as to acquire the optimal basal plane orientations for enhanced tribological performances [8]. Similarly, the possible synergism of a combination of two lubricants was reported by Gardos [9] where the synergistic effect was ascribed to the formation of oxidized layer that act as a diffusion barrier on the surface of the friction film. In this scenario metal dichalcogenides such as tungsten disulfide (WS_2) may be useful since their lubricity remains unaffected till temperatures well above ~450 °C, i.e. without any oxidative degradation [10]. Due to weak interatomic interactions and layered structure morphology the metal dichalcogenides such as tungsten disulfide are inherently able to enhance the shearing assisted mechanism of wear resistance enhancement [11]. Recently Si et al. [12] have established the effectiveness of wear performance enhancement of WS_2 and WO_3 filled paraffin nanocomposites where fivefold wear reduction by the incorporation of micron sized WS_2 and thirteenfold wear reduction due to addition of nano-WS_2 to paraffin was reported. In another independent study by Wong et al. [13] on surface and friction characterizations of WS_2 and MoS_2 containing third bodies in extreme friction contact situations such as in wheel-rail contact the use of metal dichalogenides have been found to exhibit lower decomposition temperatures in tribo-chemical reactions when compared to thermal reactions. From the Amsler friction plots it was deduced that the conversion of WS_2 to WO_3 decisively modifies the stable friction regime and contributes to the slow increase in friction. Theoretically these observations may form the basis of material design conjectures like the incorporation of such fillers into tribo-materials in other extreme contact situations such as in automotive brake linings may lead to enhanced recovery performance that theoretically demands slow increase in friction when the friction material is subjected to perform at lower temperatures. Li-na [14] while investigating the microstructure and tribological properties of WS_2/MoS_2 multi layer films have reported that multilayered films on steel surface may lead to lower friction and enhance wear resistance.

Flyash cenospheres as ceramic based cenospheres that are essentially highly inert hollow spheres comprising of silica, iron and alumina are typically produced as by product of coal combustion at ~1500 to 1750 °C through a series of complicated chemical and physical transformations. Inherently these cenospheres are highly porous, abrasive and thermally stable and hence may potentially be exploited in automotive friction industry since such censopshere filled phenolic based resin composites may give rise to composites with enhanced adiabatic potential [15,16]. Cenospheres as successful multifunctional fillers in automotive friction composite formulations have been well demonstrated though due to intrinsic compositional variations in terms of the mixture of the various metal oxides the friction performance is prone to undulations and instabilities. For example, Jaggi et al. [17,18] have reported the improved wear resistance for flyash based composites at an optimized glass fiber content of 5 wt.% based on dilution of phenolic resin. Similarly, the enhancement in the structural, thermal, mechanical and dynamic mechanical performance of cenosphere filled thermoplastic matrix based composites has also been reported [19]. In this back ground the present paper deals with the investigation on the potentially synergistic combination of cenosphere, as a sustainable multi-functional filler, and WS_2 as a high temperature lubricant, in friction composites evaluated for their fade and recovery performance following industrial standards as per the European commission for energy (ECE) norms.

2. Experiment

2.1. Fabrication procedure for molding of composites

Novolac type straight phenol–formaldehyde resin (JA10) based and cenosphere (average particle size of 40 μm) and tungsten disulfide (WS_2) filled friction composites have been fabricated. The composites were reinforced by Kevlar pulp (organic fiber), Lapinus fiber (volcanic rock fiber: RB 250), Vermiculite (Flakes) and Steel wool (metal fiber) synergistically. The detail of the compositional variation and nomenclature of the fabricated composites is shown in Table 1. The mixing schedule and molding conditions have been adopted as per standard industrial practice based on the curing characteristics of novolac resin. The processing conditions of the composites are given in Table 2. The mould cavity was supported by the adhesive coated back plate of mild steel which enables the production of brake pads after compression molding. To remove moistures and other gaseous by-products released during curing and to prevent cracking of the composites, multiple intermittent breathings were allowed during molding. The pads (with surface area of 32 cm^2) were post-cured to relieve processing induced frozen-in stresses that might originate due to improper packing or non-uniform curing of the resin.

2.2. Physical, mechanical and surface characterizations

The composites were characterized for their density by volume displacement method. Soxhlet apparatus was used to carry out acetone extraction to estimate the amount of any other organic fraction and/or uncured resin so as to identify the composition. The ash content was determined gravimetrically after roasting at about 850 °C in a muffle furnace for 6 h. The mechanical properties such as hardness (measure of resistance to indentation under loads) was measured on Digital hardness tester, cross-breaking/shear strength (to characterization of composite integrity throughout the bulk) was measured on Universal Testing Machine (model: UTN 20, S.No: 9/83-603) from Fuel Instruments and Engineering Pvt., Ltd. and following standards conforming to industrial practice,