

# Mechanical and tribological properties of phenolic resin-based friction composites filled with several inorganic fillers

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## Abstract

The calcined petroleum coke (CPC), talcum powder (TP) and hexagonal boron nitride (h-BN) were used as the friction modifiers to improve the mechanical and tribological properties of phenolic resin-based friction composites (the resin matrix was coded as PHE). Thus the composites filled with the inorganic particulates of laminar structures were prepared by compression molding. The hardness and bending strength of the friction composites were measured. The tribological properties of the composites sliding against cast iron were evaluated using a pin-on-disc test rig. The morphologies of the worn surfaces of the composites and the transfer films on the counterpart cast iron disc were analyzed by means of scanning electron microscopy, and the elemental plane distributions on the transfer films were analyzed using energy-dispersive X-ray analysis (EDXA). It was found that the friction composites of different compositions showed different friction and wear behaviors, which was highly dependent on the volume fractions of the friction modifiers in the composites. Namely, the inclusion of CPC, h-BN, and TP at a volume fraction of 10% helped to greatly increase the bending strength and wear resistance of the composites, and in these cases the coefficients of friction for the composites were ranged within 0.43–0.47. In particular, the PHE-based composite with 10% h-BN had excellent friction stability at various testing conditions and showed the best wear resistance above 125 °C, which was attributed to the formation of a compact friction film (third-body-layer) on the rubbing surface of the composite and of a durable transfer film on the rubbing surface of the counterpart cast iron. The PHE-based composite with 10% CPC showed the best wear resistance below 125 °C, which was ascribed to the same reasons mentioned above. The different actions of various friction modifiers in terms of their effects on the friction and wear behavior of the phenolic resin-based friction composites could be related to their different bonding strengths with the resin matrix and their different abilities to form friction films (third-body-layer) on the surfaces of the composites and transfer films on the counterpart cast iron surface as well.

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**Keywords:** Phenolic resin-based friction composite; Friction modifier; Mechanical properties; Friction and wear behavior; Transfer film

## 1. Introduction

As a kind of non-asbestos organic (NAO) friction materials, 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 reinforcing

fibers, abrasives, binders, fillers, and friction modifiers (solid lubricants) into phenolic resin-based friction composites for the purpose of increasing the stability and wear resistance [1–4]. And naturally, the mechanical and tribological properties of the phenolic resin-based friction composites are greatly dependent on the interactions and synergetic effects among the multiphase ingredients. In this sense, it is crucial to correctly and properly select and combine the different components so as to satisfy a number of requirements for the properties such as good wear resistance, small wear to the counterparts, stable coefficient of friction, and reliable strength at a wide range of stressing conditions of the friction materials [5,6]. Loken [7] found that crimped and chopped Kevlar® fiber together with wollastonite and dolomite contributed to greatly increasing the friction and

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wear properties of the phenolic resin-based friction materials. Sinha et al. [8] said that the addition of Kevlar® fibers to phenolic resin reduced the coefficient of friction. Gopal et al. [9,10] found that the use of Kevlar pulp and glass fiber in the friction materials lowered wear and coefficient of friction, eliminated squeal, and enhanced friction stability. It has been found that among many ingredients currently used in the composites, the inorganic particulates used as friction modifiers (solid lubricants) usually play crucial roles in determining the friction characteristics. And many investigations about the control of the friction characteristics by adding proper friction modifiers have been reported. For example, Jang and Kim. [11] found that the relative amounts of  $\text{Sb}_2\text{S}_3$  and  $\text{ZrSiO}_4$  in phenolic resin-based friction material played a crucial role in determining the wear of friction material during service. Høyer et al. [12] said that the addition of various metal sulfide solid lubricants led to much larger variation in the friction and wear behavior of resin-based automotive disc brakes. Surprisingly, as one of the most widely used traditional solid lubricants, though  $\text{MoS}_2$  has been found to considerably improve the friction and wear behaviors of various bonded solid lubricating coatings, it has detrimental effect on the wear resistance of some composites, due to the oxidation of  $\text{MoS}_2$  to  $\text{MoO}_3$  in air [13–16]. Enlightened by this observation, we anticipate that many other laminar inorganic particulates such as low cost calcined petroleum coke (CPC), which is mainly comprised of graphite particles and amorphous carbon, heat-resistant hexagonal boron nitride (h-BN), and talcum powder (TP) would also have complicated effects on the tribological properties of phenolic resin-based friction composites. With this perspective in mind, CPC, h-BN, and TP of laminar structures similar to that of graphite and  $\text{MoS}_2$  were selected as the friction modifiers for phenolic resin-based friction composites. The target phenolic resin-based friction composites filled with the friction modifiers were prepared by compression molding, using E-glass fiber as the reinforcement. The mechanical and tribological properties of the friction composites were evaluated, with the effects of the friction modifiers on the mechanical and tribological properties and the transfer film formation at the rubbing interface to be highlighted.

## 2. Experimental

### 2.1. Sample formulation and preparation

The friction composite specimens contained a binder, reinforcements, friction modifiers, fillers, etc., as shown in Table 1. The cashew-modified phenolic resin powder (Jinan Shengquan Hepworth Chemical Co. Ltd., Shandong, China) containing hexamethylenetetramine (a curing agent) was used as the matrix. The milled E-glass was used as the reinforcing fiber. The glass fiber had an average filament diameter of  $15.8\ \mu\text{m}$  and a density of  $2.55\ \text{g/cm}^3$ . The calcined petroleum coke had a grit size of  $0.05\text{--}0.50\ \text{mm}$  and a density of  $2.04\ \text{g/cm}^3$ . The talcum powder and hexagonal boron nitride were purchased from Beijing Chemical Reagent Co. Ltd.,  $\text{Al}_2\text{O}_3$  particulates used as the abrasive had a diameter of  $0.7\text{--}0.8\ \mu\text{m}$ .  $\text{CaCO}_3$  was used as the space and balance filler.

Table 1  
Compositions of the phenolic resin-based friction composites

Ingredients	Content (vol.%)
E-glass fiber	21
Phenolic resin	35
$\text{Al}_2\text{O}_3$ microparticles	4
Space filler $\text{CaCO}_3$	40–15
Friction modifiers	0–25
Calcined petroleum coke (CPC)	
Talcum powder (TP)	
h-Boron nitride (h-BN)	

The powders of CPC or TP and h-BN were added to the raw materials mixture at a volume fraction of 0%, 5%, 10%, 15%, 20%, and 25%, respectively, corresponding to the  $\text{CaCO}_3$  volume fraction of 40%, 35%, 30%, 25%, 20%, and 15%, with the contents of the other ingredients being fixed as shown in Table 1. The ingredients were mixed for 1–2 min, by stirring in a blender so as to improve the dispersion of the milled fibers and various other components in the raw materials mixture, and then compression molded to give three types of phenolic resin-based friction composites filled with different friction modifiers (the composite without any friction modifiers was coded as PHE and used for the comparative study). The fully mixed raw material ingredients were loaded into a  $175\ \text{mm} \times 65\ \text{mm} \times 37\ \text{mm}$  mold and hot pressed at  $170\ ^\circ\text{C}$  and 25 MPa for 30 min in an oven. After releasing of the pressure in the mold, the molded slab sample was post-cured at  $190\ ^\circ\text{C}$  for about 8 h in the oven. Finally the molded composite slabs were cut and ground to give the  $\phi 3\ \text{mm} \times 30\ \text{mm}$  test specimens (pins).

### 2.2. Measurement and analysis

The mechanical properties of the phenolic resin-based friction composites were determined using a universal materials tester at room temperature. The hardness of the friction composites was measured using an optical hardness tester at a Brihell Rockwell Vickers scale, according to ISO178-1993.

The friction and wear behaviors of the phenolic resin-based friction composites were evaluated using a pin-on-disc tribometer. The schematic of the pin-on-disc pair is described in detail elsewhere [17]. Briefly, a cast iron disc (pearlitic iron, 25 mm in diameter and 8 mm in thickness, HT250) was used as the lower counterpart and driven by a motor to slide against the upper composite pin (end-face contact, with the contact pressure to be  $0.71\text{--}6.4\ \text{MPa}$ , which covers the contact pressure  $3.0\ \text{MPa}$  used for the evaluation of the friction and wear behavior of braking materials using bench test) at different operating conditions. Prior to the tests, the cast iron disks were polished with 600# sand paper to a surface roughness  $R_a = 0.19\ \mu\text{m}$ , and then cleaned with acetone. The friction and wear tests were conducted at a sliding velocity of 0.2 or 0.4, 0.6, 0.8, 1.0, 1.2 m/s, a load of 5 or 7, 10, 12, 15 N, a temperature of 25 or 125, 225, 325,  $425\ ^\circ\text{C}$ .

After the friction and wear tests, the pins and disks were ultrasonically cleaned in acetone for 5 min, dried in air, and then

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