

# Design of experiments approach to the study of tribological performance of Cu-concentrate-filled PPS composites

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## Abstract

The tribological performance of copper-concentrate (CC) mineral deposit as the filler in polyphenylene sulfide (PPS) was studied as a function of the filler proportions and sliding test variables. CC is a complex mixture of CuS, Fe<sub>x</sub>O<sub>y</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and other trace materials. The design of experiments based upon L<sub>9</sub> (3<sup>4</sup>) orthogonal arrays by Taguchi was used. Sliding tests were performed in the pin-on-disk configuration against a hardened tool steel (55–60 HRC) disk. The improvement in wear resistance of PPS was considerable with the use of fillers. The lowest steady state wear rate of 0.0030 mm<sup>3</sup>/km was obtained for PPS + 20%CC + 15%PTFE composition. It was two orders of magnitude lower than that of unfilled PPS. The variations in steady state coefficient of friction with the changes in filler proportions and sliding test variables were small. The transfer film was studied by atomic force microscopy (AFM) and scanning electron microscopy (SEM). X-ray photoelectron microscopy (XPS) was used to detect chemical reactive species developed during sliding, especially in the interface between transfer film and its counterface. Wear particles and the polymer worn surfaces were analyzed by energy dispersive spectroscopy (EDS) for elemental distribution.

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## 1. Introduction

The improvement of mechanical and/or tribological properties of polymers by incorporation of particulate filler materials and short fibers has been widely studied [1]. It has been noticed that there is synergism between the filler and fiber materials when both are simultaneously present, and such hybrid composites provide tribological properties unachievable by the filler or the fiber alone [2]. An alternative to these hybrid composites would be the composites filled with minerals which consist of a complex mixture of inorganic compounds. The tribological behavior of such composites has not been studied. The interaction of these compounds with the counterface due to high pressure and heat at the sliding interface could make the tribological behavior of these composites very complex.

Studies have been carried out using a variety of inorganic filler materials in order to reduce wear and increase or decrease the coefficient of friction of polymer composites. One of the inorganic filler materials which is pertinent to this study is CuS. The reduction in wear rate with the addition of micro CuS filler has been reported in the case of high-density polyethylene [3], polyetheretherketone [4], polytetrafluoroethylene [5], and polyamide 11 [6]. Yu and Bahadur [7] reported that the wear rate of PPS was significantly reduced by the addition of 35 vol% CuS. In addition, the synergism effect was also reported when CuS and short carbon fibers as reinforcement material were used together in nylon [2]. It was also shown that the development of a thin and uniform transfer film on the counterface during sliding was associated with the reduction in wear rate.

In addition to filler type and its proportions, sliding variables such as counterface roughness, sliding speed, and applied load have been known to affect the tribological behavior of polymer composites. As for the sliding speed,

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the faster the sliding speed, the higher was the wear rate in the case of ultra-high molecular weight polyethylene, polyphenylene oxide, polyetheretherketone, and polytetrafluoroethylene where sliding speed was varied from 0.001 to 10.0 m/s [8,9]. However, the coefficient of friction did not follow the same pattern as observed for the wear rate. A high sliding speed of 2.0 m/s caused thermal softening in case of the polymers [10]. Bahadur and Gong [11] showed that wear rate of nylon and nylon filled with 35 vol% CuS increased when the counterface roughness was increased from 0.04 to 0.30  $\mu\text{m}$  Ra. It was so because of the incomplete coverage of transfer film on the rougher counterface. The wear of polymer composites was found to be closely related with sliding variables, but no regular pattern between the variables was found to exist.

Studies showing the relationship between transfer film formed on a counterface during sliding and wear behavior of polymer composites have been reported [2–7]. Yu and Bahadur [7] reported that wear resistance was substantially increased when transfer film was thin and smooth. Furthermore, poor cohesion and adhesion of the transfer film to the counterface contributed to increased wear rate.

## 2. Experimental details

### 2.1. Materials

Polyphenylene sulfide (PPS) was used as the base material due to its high strength and high-temperature capability. The latter was important because sliding tests were run at high sliding speeds. Another high temperature polymer suitable for high sliding speed sliding could be PEEK but PPS was selected for this investigation because the tribological studies on PPS are comparatively fewer than on PEEK. Furthermore, it was a part of the continuing tribological studies with many other fillers in our laboratory. PPS in the form of powder was supplied by the Philips Chemical Company. PTFE (FLUON<sup>®</sup> Grade G163) in powder form was supplied by Asahi Glass Fluoropolymer Co. It was used as a solid lubricant in PPS composites.

The mineral filler used in this study was copper-concentrate (abbreviated as CC) which is composed of a mixture of inorganic compounds and iron oxides. The major constituents in this mineral are CuS, FeO, Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, CaO, and SiO<sub>2</sub>. The proportions of these and other constituents are given in Table 1. The filler used was in the form of powder of 45  $\mu\text{m}$  maximum size.

### 2.2. Sample preparation

PPS, CC, and PTFE were dried at 125 °C before weighing to prepare the specimens with desired proportions. The mixture was mechanically blended in an ultrasonic bath of acetone. It was dried and compacted in a cylindrical mold 6.35 mm in diameter and 11.00 mm long. The specimen was compression molded at 310 °C as

Table 1  
Constituents of copper-concentrate

Constituents	Contents (%)
CuS	46
Fe <sub>2</sub> O <sub>3</sub>	24
FeO	13
SiO <sub>2</sub>	7.3
Al <sub>2</sub> O <sub>3</sub>	2.3
CaO	1.1
MgO	0.46
MoS <sub>2</sub>	0.02
Pb	0.40
As	0.50
Zn	0.35
Ag	65.5 grams/ton
Au	4.0 grams/ton

described elsewhere [12]. A total of nine specimens with the compositions given in Table 2 were prepared.

### 2.3. Pin-on-disk sliding tests

Pin-on-disk sliding tests were performed on a hardened tool steel counterface (55–60 HRC). The specimen for wear test was 6.2 mm in diameter and 10 mm long. The test surface was finished by abrasion, washed, and dried. As shown in Table 2, wear tests were performed at the sliding speeds of 0.5, 1.0, and 1.5 m/s. A nominal contact pressure of 0.65 MPa was used. The counterfaces were finished by abrasion to provide three roughnesses. The combinations of the sliding speed and counterface roughness for each composition are given in Table 2. Sliding tests were performed over a distance of 21.6 km regardless of the sliding speed. The long duration tests ensured steady state wear which is emphasized in this study. The mass loss was measured with an accuracy of 10  $\mu\text{g}$  at the sliding distances of 1.8 and 3.6 km for the first two data points and at every 3.6 km thereafter. It was converted to volume loss using the density of the specimen. The coefficient of friction was also measured using strain gages which were mounted on the loading arm. Three wear tests for each composition were carried out and were averaged. The results were within a scatter range of  $\pm 15\%$  for wear and  $\pm 10\%$  for friction.

### 2.4. Design of experiments

In order to investigate the tribological behavior of the composites with the variables used in this study, design of experiments approach by Taguchi was employed. In this study, full factorial sliding tests with four parameters and three levels require 81 (3<sup>4</sup>) tests. However, only nine experiments are needed with the use of the simplified orthogonal arrays designed by Taguchi, and this provides a great advantage over the full factorial experiment as described elsewhere [12,13]. The variables included were sliding speed, counterface roughness, and CC and PTFE

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