



Impact of counterpart materials and nanoparticles on the transfer film structures of polyimide composites

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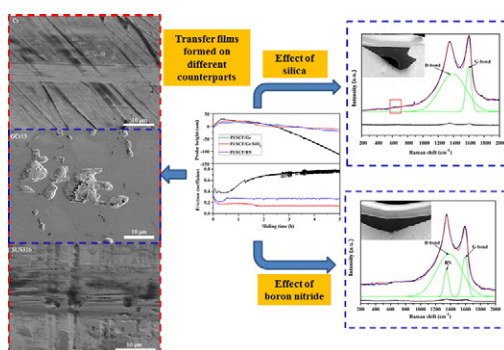
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HIGHLIGHTS

- Oxidation resistance of counterparts exerts an important role in transfer films of polyimide composites.
- Silica and hexagonal nitride boron nanoparticles inhibit tribo-oxidation of counterpart metals.
- Inclusion of nanoparticles in polyimide composites leads to formation of lubricating transfer film.

GRAPHICAL ABSTRACT



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ABSTRACT

The nanostructures of the transfer films of polyimide (PI) composites generated when slid against various metallic counterparts including standard bearing steel, electroplated chromium coating (Cr), and stainless steel were comprehensively investigated. Moreover, the effect of h-BN and amorphous SiO₂ nanoparticles on the transfer film structures of the conventional composite filled with short carbon fibers and graphite were studied. It was identified that the composition of the counterparts and nanoparticles' structure exert important effects on transfer film formation. When the conventional composite rubs with the steel counterparts, obvious tribo-oxidation occurs and therefore retards material transfer. However, the composite shows a much improved tribological performance when slides against Cr due to formation of a carbonaceous transfer film. Both kinds of nanoparticles significantly mitigate tribo-oxidation and greatly enhance the tribological performance. Nevertheless, the two different kinds of nanoparticles lead to distinct transfer film structures. High resolution transmission electron microscopy analyses of transfer films reveal that tribo-sintering of nano-SiO₂ occurs, whereas h-BN particles are crushed and uniformly mixed with wear products. Moreover, h-BN transforms to c-BN probably owing to the high temperature and stress on the rubbing interface. This work can provide guidance for the formulation of best matching composites according to metallic counterparts.

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1. Introduction

It is well recognized that the formation of a transfer film due to complex tribo-physical interactions and tribo-chemical reactions occurring on the interface plays an important role in the tribological

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performance of polymer-based materials when rubbing with metallic counterfaces [1–5]. A high-performance transfer film effectively reduces friction and wear and thus enhances the tribological properties by preventing the direct contact of the sliding pair and providing efficient lubricating action [6–9].

Great attention has been paid to the formation and function mechanisms of the transfer films generated on metallic counterparts [10–12]. In particular, the effects of polymer matrix and composite formulation on transfer film formation were investigated [13,14]. Gong et al. [2] and Gao [7] revealed that fragmentation of Polytetrafluoroethylene (PTFE) molecular chain and thereafter chelating reaction of the free radical with steel counterface dominated the transfer film formation. It was also demonstrated by numerous works that when certain types of polymer matrix, e.g. Polyformaldehyde (POM) [15], Polyamide (PA) [16] and Ultra High Molecular Weight Polyethylene (UHMWPE) [17], were slid against a metallic counterpart, a homogeneous transfer film usually formed because of their good “self-lubrication” properties. The layer structure of solid lubricant fillers gives rise to the formation of a lubricant film on the counterface [18,19]. Reinforcing fillers such as carbon fibers, glass fibers and aramid fibers significantly enhance the load-carrying capability and improve the abrasion resistance of the polymer matrix [20]. Moreover, the high stress and temperature occurred on the interface between reinforcing fillers and metallic counterface can trigger various tribo-physical and chemical reactions [1,21,22].

Numerous studies identified that nanoparticles such as Al_2O_3 , CuO and CuS could react with polymer molecules or metal surfaces and produce different chemical substances which determined the composition of the transfer film [10,22–27]. Harris et al. [10] reported that Al_2O_3 nanoparticles can react with fragmented PTFE molecules and the counterpart steel. The authors revealed that these reactions were of great importance for forming a robust and lubricating transfer film on the steel counterface. Chang et al. [28,29] reported that TiO_2 nanoparticles added into epoxy-based conventional composites led to the formation of a homogeneous transfer film on a standards bearing steel. The authors characterized the mechanical properties of the transfer film using nano-indentation technique [28]. Nevertheless, it is noteworthy that transfer film is usually extremely thin and characterization of its nanostructure is not accessible without special and expensive preparation techniques. Although many publications have shown the importance of transfer films of polymer composites generated during dry friction applications, only few results provide the information about their nanostructures.

Tribology is a system property governed by both sides of the sliding pair. It is believed that the transfer film structure is influenced not only by the formulation of polymer but also by the physical and chemical properties of the counterparts. However, to date a majority of investigations on dry sliding transfer films of polymers considered only steels as counterparts, e.g. standard bearing steel [30–32] and medium carbon steel [33]. In fact, a variety of metal counterpart materials are being employed in dry sliding applications, design of best matching tribo-pairs becomes an important issue. However, only a few researches reported the effect of counterpart materials on the tribological performance of polymer [34,35]. In particular, the impact of counterpart materials on the transfer film structures of polymer composites was rarely reported. Thus, a solid understanding of the effect of counterpart materials on the formation and function mechanisms of the transfer film is of both fundamental and practical interests.

Polyimide (PI) is a high-performance polymer exhibiting great thermal stability and excellent mechanical properties. Thus, PI becomes an attractive matrix material for developing high-performance tribo-composites exposed to severe running conditions for applications in the fields of aerospace and transportation [36–39]. However, the effect of counterpart materials on the tribological mechanisms of PI composites has not been investigated yet. In this work, a variety of techniques were applied to comprehensively characterize the micro/nanostructure of transfer films formed on different counterparts after sliding with PI

composites. Three kinds of typical counterpart materials such as standard bearing steel, stainless steel and electroplated chromium coating (Cr) are investigated, which are widely used as shaft materials in practical applications. We firstly studied the effect of counterpart materials on transfer film structure and tribological behavior of a conventional PI composite filled with short carbon fibers (SCF) and graphite. Moreover, the effect of amorphous silica and hexagonal boron nitride (h-BN) nanoparticles added into the conventional composite on the nanostructures of the transfer films is studied. The main goal of this study is to gain insight into the dependence of the transfer film structure of PI composites on counterpart materials and the nanoparticle types. The outcome of this work will provide information for formulating polymer composites according to counterpart materials.

2. Materials and method

2.1. Material preparation

Thermoplastic PI was supplied by Shanghai Synthetic Resin Institute (Shanghai, China) in form of powders (YS-20, particles size $<75 \mu\text{m}$) with the thermal deformation temperature of 239°C (GB1634). Polyacrylonitrile (PAN)-based carbon fibers supplied by Nantong Senyou Carbon Fiber Co., Ltd., China, were ultrasonically cleaned with acetone for 2 h and then added into PI polymer matrix as reinforcing fillers. The short carbon fibers were $20\text{--}45 \mu\text{m}$ in length and approximately $7 \mu\text{m}$ in diameter. Graphite flakes (RGB390TS, Superior graphite, particles size $\approx 4 \mu\text{m}$) were used as internal lubricants. Amorphous nano-silica (SiO_2) with an average diameter of 20 nm was provided by Nanjing Emperor Nano Material Co., Ltd., China. h-BN nanoparticles with an average diameter of 120 nm were supplied by Shanghai St-Nano Science&Technology Co., Ltd., China.

SCF, graphite, SiO_2 or h-BN were dispersed into PI polymer matrix using high speed crusher. The abbreviated forms and the specific composition of various PI composites were listed in Table 1. The composites were prepared by means of hot press molding technique. The mixtures were compressed and heated from room temperature to 375°C in a mold with a dimension of $50 \times 60 \times 10 \text{ mm}^3$. The pressure was held at 14 MPa for 2 h to allowing full compression sintering with intermittent deflation. At the end of each run of compression sintering, the specimens were cooled to room temperature in air and cut into $4 \times 4 \times 12 \text{ mm}^3$ pins for testing.

2.2. Tribology tests

Dry friction tests were performed at room temperature using a Pin-On-Disc test rig (POD, TRM-100, Wazau, Germany) and the schematic of the contact configuration of the sliding pair is given in Fig. 1. A rotating polymer pin with a contact surface of $4 \times 4 \text{ mm}^2$ was pressed against a stationary counterface. The diameter of the friction track was 33 mm . Three types of metallic counterparts, i.e. GCr15 (GB/T 18254-2002, standard bearing steel), Cr and SUS316 (GB 9944-1988, stainless steel) were used in the test. The Cr counterpart was obtained by electroplating chromium on the GCr15 disc at Lanzhou Flight Control Co., Ltd., China and the coating thickness was $20 \mu\text{m}$. The coating compositions were analyzed by using Energy Dispersive X-ray Spectroscopy (EDS) instrumented onto the Scanning Electron Microscope (SEM) (JSM-5600LV) (Fig. S1). The chemical compositions of GCr15, Cr and SUS316 were listed in Table 2. The Vickers hardness of SUS316, GCr15 and Cr was measured

Table 1
Abbreviated forms and specific compositions of PI specimens studied (vol%).

| Abbreviated form | PI | SCF | Graphite (Gr) | SiO_2 | BN |
|---------------------------|----|-----|---------------|----------------|----|
| PI/SCF/Gr | 82 | 10 | 8 | 0 | 0 |
| PI/SCF/Gr/ SiO_2 | 80 | 10 | 8 | 2 | 0 |
| PI/SCF/Gr/BN | 80 | 10 | 8 | 0 | 2 |

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