



# Synergism of several carbon series additions on the microstructures and tribological behaviors of polyimide-based composites under sea water lubrication



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## ABSTRACT

The effects of several carbon series additions including graphite (Gr), carbon fiber (CF) and carbon nano-tube (CNT) on the microstructures and tribological behaviors of polyimide-based (PI-based) composites under sea water lubrication were investigated systematically. Results showed that the incorporation of any filler improved the wear resistance of polyimide (PI) under sea water lubrication, but did not decrease the friction coefficient. Especially the combined incorporation of 10%Gr, 10%CF and 5%CNT (in volume) was the most effective in improving the anti-wear properties of PI. This suggested that there existed a synergetic effect among the three carbon series additions on improving the wear resistance of PI. During the friction and wear process, the carbon additions played different roles in improving the wear resistance of PI-based composites. CF with high compressive strength can carry the main load applied on the sliding surfaces to inhibit the wear of PI matrix. CNT can decrease the stress concentration around CF and further protect CF from being broken. Gr in the form of much thinner layer can not only improve the loading capacity, but also play the same role of CNT to avoid CF carrying too much load. More importantly, Gr, CF and CNT worked synergistically to condense the microstructure of PI-based composite and ameliorate the interfacial combination between all fillers and PI matrix, which well explained why the PI–10%Gr–10%CF–5%CNT composite had excellent tribological properties, even under heavy load or high sliding speed.

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## 1. Introduction

Polymer and its composites have been widely used as dry sliding materials, in particular as alternatives to metal tribomaterials operating in water or other corrosive mediums, as a result of the attractive advantages of self-lubrication and superior cleanliness and excellent corrosion resistance [1–4].

PI, a famous engineering thermalplastic, has linear or cyclic imide units and aromatic groups, which provides many extraordinary characteristics, i.e., high mechanical strength, good thermal stability, chemical inertness, self-lubrication, superior corrosion resistance, etc. [5,6]. Therefore, PI might be applied in friction components or systems operating in different lubricant conditions. i.e., dry sliding, pure water lubrication, oil or other corrosive mediums. Nevertheless, long run-in stage, high friction coefficient and poor wear resistance of neat PI restricts its application in the field of tribology [7,8]. Therefore, many previous researchers have taken

many different approaches to improve the friction-reducing and wear resistant properties [9–11]. One way is to increase the compressive strength, loading-capacity and other mechanical properties by filling reinforcement additions, i.e. CF, CNT, metallic and ceramic particles etc. Another way is to decrease the shear stress and friction force on the sliding surfaces during the friction and wear process by the incorporation of lubricating additions, i.e. Gr, PTFE and MoS<sub>2</sub>.

Yet, it is difficult to achieve polymer-based composites with outstanding tribological properties by the means of filling only one kind of filler. According to many previous studies [9,12–20], the combined incorporation of various fillers can greatly improve the friction and wear properties of materials because of synergistic effect of different fillers. Xian et al. [9] have found a synergistic effect of nano-TiO<sub>2</sub> and Gr on the tribological performance of epoxy matrix composites, and this synergistic effect is attributed to effective Gr transfer films formed on sliding pair surfaces and the reinforcing effect of the nano-TiO<sub>2</sub>. Wang et al. [18] have found that the combined incorporation of CF and Gr and nano-SiO<sub>2</sub> into PI matrix greatly improve the tribological properties of PI at heavy load and

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high sliding speed under dry friction. Xie et al. [19] have investigated the influences of potassium titanate whisker (PTW) and CF on the friction and wear behavior of polyetheretherketone composite under water lubricated condition and obtain that PTW and CF work synergistically to enhance the wear resistance of the hybrid reinforced PEEK composite under water lubrication. Zhang et al. [20] have found that the monodispersed nano-SiO<sub>2</sub> can further improve the friction-reducing and wear resistant properties of epoxy composites filled with CF and Gr lubricants significantly, because the nanoparticles promote the formation of a homogeneous transfer film on the surface of counterpart. Furthermore, Friedrich et al. [16] have systematically reviewed the effects of various fillers on the sliding wear of polymer composites. Unfortunately, these investigations mainly correlate the effect of different fillers on the friction and wear behaviors of polymer composites under dry friction or pure water condition; but, few reports are currently available about the tribological properties of polymer composites under sea water lubrication, not to mention the synergistic effect of the fillers therein.

In this study, various PI-based composites reinforced with Gr, CF and CNT were prepared by hot pressing technique, and the main purpose was to investigate the synergism of these fillers on the microstructures and tribological behaviors of PI-based composites under sea water lubrication. Besides, the effects of load and sliding speed on the tribological properties of PI-based composites under sea water lubrication were studied as well. Hopefully, this study can provide some guidance for designing and developing PI-based composite with excellent wear resistance suitable for sea water environment.

## 2. Experimental details

### 2.1. Materials and specimens

PI (YS-20) powder (particle size of <75 μm, density of 1.38 g/cm<sup>3</sup>) was commercially obtained from Shanghai Synthetic Resin Institute (China). Gr (particle size of 45–500 μm, density of 2.25 g/cm<sup>3</sup>) was provided by Qingdao Haida Graphite Co., Ltd. (China). And PAN-based CF (HT, average diameter of 7 μm, length of 28–56 μm, density of 1.77 g/cm<sup>3</sup>) was provided by Nanjing Fiberglass R&D Institute (China). CNT (diameter of 10–20 nm, length of 30 μm, density of 2.10 g/cm<sup>3</sup>) was produced by Chengdu Organic Chemicals Co., Ltd., Chinese Academy of Sciences (China). And Fig. 1 exhibits the SEM micrographs of Gr, CF and CNT.

PI-based composites were prepared by means of hot pressing technique. Various volume fractions of Gr (5%, 10% and 20%), CF (5%, 10% and 20%) and CNT (3%, 5%, 7% and 10%) reinforced PI-based composites have been prepared firstly, which are to determine the optimized contents of CNT, CF and Gr. Based on this, the combination of any two kinds or three kinds of fillers at their optimized contents reinforced PI-based composites have been fabricated to further investigate the synergistic effect of carbon series additions. Specifically, the preparation method was introduced as follows: at first, the powders of PI and various fillers in the appropriate proportion were mixed in the acetone with the assistance of ultrasonic dispersion, followed by filtrating and drying. Then the mixed powders were compressed under the pressure of 30 MPa and heated to 347 °C for 60 min in the mould. After naturally cooled below 100 °C and released from the mould, the PI-based composite with a dimension of 19 mm × 12 mm × 12 mm was obtained.

### 2.2. Preparation of sea water

Sea water was prepared according to ASTM:D 1141-98 (2013) Standard practice for the preparation of substitute ocean water.

Table 1 shows the chemical composition of sea water, where the gross concentration employed in the standard is an average of many reliable individual analyses.

### 2.3. Friction and wear tests

The friction and wear tests were conducted on a ring-on-block test rig. The schematic contact diagram of the frictional couple is shown in Fig. 2. The upper block was polymer specimen, the lower ring with a size of Ø 49.22 mm × 13.06 mm was made of 316 stainless steel. And the chemical composition was as follows: ≤0.08% C, ≤1.00% Si, ≤2.00% Mn, ≤0.035% P, ≤0.03% S, 10.0–14.0% Ni, 16.0–18.5% Cr, 2.0–3.0% Mo, and balance Fe, at weight fraction. The Vickers-hardness of 316 stainless steel was HV 347.

Sliding was performed with a period of 120 min, sliding speeds of 0.5 m/s and 1 m/s, normal loads of 200 N, 400 N and 600 N. Before each test, the surfaces of specimen and counterpart ring were polished with 1000 grit paper to reach a surface roughness of about Ra 0.1 μm, and then cleaned with acetone. The sea water lubrication between the sliding surfaces was realized by continuously dropping sea water onto the sliding surface at a rate of 100–105 drops per minute (300–315 mL/h). The friction coefficient was continuously recorded by an on-line data acquisition system attached to the tester. And the final reported friction coefficient was the average value during the whole friction test. After the friction test, the width of the wear track on the specimen block was measured using an optical microscope with an accuracy of 0.01 mm. Then, the wear volume loss *V* of the blocks was calculated from the relationship:

$$V = B \left[ R^2 \arcsin \frac{b}{2R} - \frac{b}{2} \sqrt{R^2 - \frac{b^2}{4}} \right] \quad (1)$$

where *V* is the wear volume loss (mm<sup>3</sup>), *B* is the width of the block sample (mm), *R* is the radius of the steel ring (mm), and *b* is the width of the wear track on the block specimen (mm). The specific wear rate *K* (mm<sup>3</sup>/N m) was calculated as below:

$$K = \frac{V}{L \cdot d} \quad (2)$$

where *d* is the sliding distance (mm) and *L* is the load (N). Three repeated friction and wear tests were carried out for each specimen, and the average of the tests was reported.

The worn surfaces of PI and its composites were observed using a JEM-5600LV scanning electron microscope (SEM). In order to increase the resolution for the SEM observation, the tested polymer samples were plated with gold coating to render them electrically conductive. Besides, the microstructures of PI and its composites were examined by observing the FESEM morphology of their fractured surfaces as well.

## 3. Results

### 3.1. Friction and wear properties of PI-based composites under sea water lubrication

To determine the optimized volume fractions of Gr, CF and CNT, the effects of the contents of Gr, CF and CNT on the tribological properties of PI-based composites have been investigated systematically. And Table 2 exhibits the friction and wear properties of PI-based composites sliding against 316 steel under sea water lubrication. It can be found that when the contents of Gr, CF and CNT are 10%, 10% and 5%, the corresponding PI-composites have much better tribological properties compared to other composites with other contents of Gr, CF or CNT. Based on this, the synergistic

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