



# Preparation and tribological properties of polyimide/carbon sphere microcomposite films under seawater condition

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## ABSTRACT

The polyimide (PI)-based microcomposites reinforced with carbon sphere were prepared by situ polymerization and mechanical, thermal and tribological properties of PI/carbon sphere microcomposites were investigated. The results showed that the addition of carbon sphere greatly enhanced mechanical and thermal property of PI. In addition, PI/carbon sphere microcomposites had lower friction coefficient under seawater condition than other conditions because of better lubricating effect of seawater. Particularly, 0.7 wt% PI/carbon sphere microcomposite had lowest friction coefficient under seawater lubrication. However, 0.7 wt% PI/carbon sphere microcomposite showed higher wear rate under seawater lubrication than other conditions because of structure defect of microcomposites.

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## 1. Introduction

The worsening problem in environmental pollution has prompted the development of water hydraulic transmission technology. In order to make water hydraulic transmission work under seawater situation, the research of high hardness, anti-corrosion and wear resistance materials has become an indispensable important link of water hydraulic components research. However, the physical and chemical properties of seawater, such as the strong corrosion, high salinity and electrical conductivity, result in numerous problems. Therefore, it is necessary for us to develop some new materials which can meet the requirement of seawater [1–4].

As newly born polymer material, polymer composite exhibits excellent physical and chemical properties that include light weight characteristics coupled with high strength, thermal stability, chemical resistance, flexural performance, relatively easy processing, self-lubricating capability, small friction coefficient, excellent anticorrosion property corrosion resistance and outstanding wear resistance [5,6]. Polyimide (PI) has very extraordinary comprehensive performance, such as excellent mechanical properties, high dielectric properties, prominent thermal endurance, acid-resistant and alkali-resistant properties, and good friction lubrication characteristics under low or high temperature [7,8]. However, it cannot be widely used as lubricant material because of its limited tribological

performance. Usually, an improvement in the tribological behavior of polymers is expected as a result of introducing fillers into the matrix material. Recently, international scholars have conducted numerous studies on the friction and wear characteristics of PI composites reinforced fillers. Zhao fabricated the aramid fibers-reinforced polyimide composites filled with talc by means of a hot press molding technique and comparatively investigated mechanical and tribological behaviors. The results showed that the elastic modulus of the composites increased with an increase of the talc, but the impact intensity and loss factor decreased. Besides, the coefficient of friction decreased with the increase of the talc content [9]. Song provided some useful information on the tribological performance of thermoplastic polyimide (TPI) reinforced with rigid glass fillers of different shapes and sizes under dry, water, and oil lubrication conditions and found that fiber-reinforced TPI has favorable wear-resistance due to its shape [10].

Carbon spheres, which are one of allotropic substance of carbon [11], are often used as a template preparation of hollow spherical materials owing to the nature of the surface easily changeable. Carbon spheres possess small density, large specific surface area and chemical stability and other merits. Therefore, they also can be applied in the area of electrochemistry to be electrode material because of good conductivity and stability [12,13]. Although carbon spheres have been widely studied in the many areas [14–18], the tribological studies of carbon spheres are scarce. Recently, neat C<sub>60</sub> had already been shown to have a fairly low friction coefficient at least under certain conditions [19]. Pozdnyakov investigated the sliding friction and wear characteristics of pyrimidine-containing polyimide (PI) coatings and PI–C<sub>60</sub> composite. The result showed

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that the introduction of C<sub>60</sub> could improve further the wear characteristics of the coatings down to specific wear rates below  $2 \times 10^{-7} \text{ mm}^3/\text{Nm}$  and suggest that the role of the C<sub>60</sub> molecules in reducing the wear of composite coatings is due to the interactions between C<sub>60</sub> and the PI [20]. Due to C<sub>60</sub> which is a kind of carbon spheres, the tribological behavior of polymers also might be improved through the introduction of carbon spheres into a mechanically and thermally stable polymer, such as PI.

Inspired on the above research, it can be seen that polymer composites reinforced with carbon spheres could make a potential application in the tribological research. However, they are concentrated in the field of dry friction. The reports on the tribological behaviors of polymer composites reinforced with carbon spheres under seawater environment have been hardly reported so far.

In this study, PI/carbon sphere microcomposite films have been prepared by the method of situ polymerization. The main objective was to investigate the tribological properties of PI/carbon sphere microcomposite films by a universal microtribotester under dry friction, pure water lubrication and seawater lubrication conditions. Anti-wear and friction-reducing mechanisms of PI/carbon sphere microcomposites have also been discussed. It is expected that this investigation can provide some valuable information to utilize the polymer composites in seawater circumstances.

## 2. Experimental section

### 2.1. Materials

Glucose, 4,4'-oxidianiline (ODA), Pyromellitic dianhydride (PMDA), N,N-dimethylacetamide (DMAC), and ethanol were commercially obtained from East Instrument Chemical Glass Co., Ltd., China. Natural seawater sourced from the Qinghuangdao sea area of Bohai was used as lubricating additive in the tribological experiments. The pH and salinity were 7.2 and 2.983%, respectively.

### 2.2. Preparations of carbon sphere

Carbon sphere was prepared by a hydrothermal method [21]. Glucose (7.927 g) was dissolved in distilled water (80 ml) with the aid of ultrasonication at RT. Then, the above mixture was reacted at 180 °C for 10 h. After the reaction, the obtained products were filtered and washed with deionized water for several times. Finally, the above products were dried in a vacuum oven for 48 h at 80 °C to obtain carbon sphere.

### 2.3. Preparation of polyimide/carbon sphere microcomposite films

The synthesis process of PI/carbon sphere microcomposite films was shown in Fig. 1. To begin with, different concentrations of carbon sphere were dispersed in DMAC (70 ml) under sonication for 6 h at room temperature, respectively. Then 5 g ODA was added into above different concentrations of carbon sphere mixture and stirred vigorously under nitrogen in the ice bath for 1 h, respectively. After ODA was dissolved in solution, 5.559 g PMDA was respectively added into mixtures and mixtures were stirred at room temperature with a nitrogen purge for 1 h so as to output the poly(amic acid)/carbon sphere. The precursor mixture solutions were cast onto a clean and dry glass substrate, baked in a vacuum oven at 80 °C for 6 h to remove air bubbles and then heated at 70 °C for 2 h at 100, 150, 200, and 300 °C for 30 min to completely achieve thermal imidization. The heating rate was 2 °C/min, which resulted in the PI/carbon sphere microcomposite films.

After the above high-temperature a thermal imidization process, PI/carbon sphere microcomposites were obtained and were not stick onto the glass substrates. Then we took PI/carbon sphere

microcomposites down directly. In order to test the tribological properties, we fixed the PI/carbon sphere microcomposites on the glass slide. In order to test the tensile properties, we prepared PI/carbon sphere microcomposites into the horseshoe shape by mold. Then, we used the horseshoe shape PI/carbon sphere microcomposites for tensile testing directly. The prepared PI/carbon sphere microcomposite films showed smooth surface by visual inspection and had high degree of the flexibility, as shown in Fig. 2. Besides, the thickness of the PI/carbon sphere microcomposite films was about 0.09 mm.

### 2.4. Evaluation of the tribological behavior

A universal micro-tribotester (UMT-2, Center for Tribology Inc., USA) was used to evaluate the friction and wear behavior of PI/carbon sphere microcomposites under dry sliding condition, pure water condition and seawater lubrication condition. The tribological model of PI/carbon sphere microcomposite film under seawater lubrication was shown Fig. 3. Under rotational motion, friction and wear tests were conducted at a rotating speed of 0.1569 m/s, under a constant load of 3 N, with the test duration of 30 min. Diameter of the steel ball was 1.6 mm. The rotating radius was 0.5 cm. Before each test, steel balls were cleaned with acetone followed by being dried. The wear volume loss  $V$  (mm<sup>3</sup>) was calculated by using the following equation [22]:

$$V = 2\pi Rbh \quad (1)$$

where  $R$  is the radius of sliding wear scar (mm),  $b$  is the width of the wear scar (mm), and  $h$  is the depth of the wear scar (mm). At the end of each test, the specific wear rate  $K$  [mm<sup>3</sup>/(Nm)] of PI/carbon sphere microcomposites was calculated according to the formula [23]:

$$K = \frac{V}{Ld} \quad (2)$$

where  $L$  and  $d$  are the normal load (N) and sliding distance (m) of the wear test. The wear test was repeated three times with different contents of PI/carbon sphere composite. The friction coefficient was recorded automatically by a strain gauge. The depth of the wear scar of the PI/carbon sphere microcomposites was measured with a three-dimensional profiler. The wear scar width values were obtained from images taken by a Leica DM 2500 M optical microscope.

### 2.5. Characterization

Fourier transform infrared (FTIR) spectra of carbon sphere, PAA, PI and PI/carbon sphere were obtained using a Nicolet Protégé-460 FTIR spectrophotometer between 4000 and 500 cm<sup>-1</sup>. The crystalline structures of glucose, carbon sphere, PI and PI/carbon sphere were investigated with XRD using a Shimadzu XRD-6000 equipped with a Cu K $\alpha$  radiation ( $\lambda=0.154 \text{ nm}$ ) source at 40 kV and 30 mA with the  $2\theta$  range from 5–90°. Tensile test of the film samples was studied using an Instron 4204 universal testing machine with a test speed of 5 mm min<sup>-1</sup>. Tensile test was tested three times for every content of PI/carbon sphere composite. The scanning electron microscope (SEM) (JEOL JSM 6700F) was used to observe the morphology of carbon sphere and PI/carbon sphere. Thermogravimetric (TGA) analyses of PI and PI/carbon sphere were performed with a Du Pont TGA 2900 analyzer from 30 to 800 °C in N<sub>2</sub> at a heating rate of 10 °C/min<sup>-1</sup>. The thermal conductivity of PI and PI/carbon sphere microcomposite films at room temperature was measured by a thermal conductivity tester (NETZSCH LFA 457). The thermal conductivity of each sample reported in this work was the average value of five measurements. Seawater absorption of PI and PI/carbon sphere microcomposite

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