

## Tribological Behaviors of PTFE Composites Filled with Surface-Modified Nano-Serpentine under Natural Seawater Lubrication

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**Abstract** — As a new developing sub-field of tribology, marine tribology is significant. In order to find a material with good tribological performance in seawater, in this paper a surface-modified nano-serpentine particles with a silane coupling agent (KH550) were filled into a Poly Tetra Fluoro Ethylene (PTFE) matrix to prepare PTFE/serpentine composites. The water absorption and hardness of the composites were investigated. Tribological tests of the composites against AISI 1045 steel were conducted using a MMU-5G friction and wear tester under natural seawater lubrication. The worn surface of the PTFE/serpentine composite and the AISI 1045 steel ring were characterized using a scanning electron microscope. The results indicate that the surface of the serpentine powder modified using KH550 has the characteristics of a silane coupling agent, which facilitates a better combination between the serpentine powder and the PTFE matrix. The water absorption of the PTFE/serpentine composite in seawater was extremely low, and the hardness of the composite hardly changed. In the tribological tests, the wear rate of the PTFE composite was one order lower than that of the pure PTFE. The friction chemical reactions occurred on the surface of the steel ring, and the transfer film and seawater lubrication film were the dominant factors that resulted in the excellent tribological performance of the PTFE composite under seawater lubrication.

**Keywords** - Sliding wear; Surface topography; Polymer-matrix composite; Tribochemistry

### I. INTRODUCTION

The development of marine science depends on the research and advancement of marine-specific materials. Particularly, tribological problems in marine environments are one of the challenges that marine materials encounter [1]. Compared with metallic materials [2] and inorganic non-metallic materials, polymers and polymer-based composites have been widely used in marine equipment because of their excellent self-lubricating property, low density, high strength, capability of embedding grit or sand and wide temperature adaptability [3-5]. Polytetrafluoroethylene (PTFE), one type of polymer material, has been extensively applied in engineering materials due to its excellent corrosion resistance, low friction coefficient and relatively low water absorption [6-8]. Previous studies have indicated that the lubricating state, plasticization of absorbing water, corrosion of contact surface, and cooling effect of lubricating media are important factors that affect the tribological behaviors of a polymer [9, 10].

Wang et al. [11] researched the tribological behavior of PTFE against GCr15 steel under seawater lubrication. They determined that the corrosive properties of seawater and the increase in the surface roughness of the GCr15 resulted in an increase in the wear rate of the PTFE. Jia et al. [12] investigated the friction and wear properties of a PTFE/CF composite under dry and water-lubricated conditions and determined that due to the cooling and lubricating properties of water, both the friction coefficient and the wear rate of

the PTFE/CF composite under water lubrication were lower compared to those under a dry environment. Chen and Wang et al. [13, 14] researched the tribological properties of PTFE/PI and PTFE/CF composites under seawater lubrication and determined that the tribological behaviors of the PTFE and its composites were not affected by plasticization because their hardness remained nearly unchanged after being immersed in seawater for 24 h. Ming Der Ger et al. [15] studied the performance of Ni-P-PTFE on water lubrication conditions, and deemed that the friction coefficient and wear are strongly related to the wettability of coupling material. Additionally, Borruto et al. [16] indicated that by coupling a hydrophilic and a hydrophobic material, such as PTFE-based composite/steel, the friction coefficient and wear rate with water lubrication were lower compared to those with oil lubrication.

The application of PTFE is limited by its disadvantages, such as high wear, poor compressive strength and low hardness. Therefore, it is necessary to fill the PTFE with different types of fillers and reinforcing agents, such as metallic materials (Cu, Pb, Ni), inorganic materials (CF, EG), organic materials, nanomaterials (nano-TiO<sub>2</sub>, MWCNTs, nano-SiC) and others [17-24], to significantly improve the wear resistance of pure PTFE. Jia et al. [3, 25] studied the tribological behavior of a PTFE/serpentine composite against steel under dry friction and determined that the addition of serpentine improved the wear rate of the PTFE due to the serpentine's distinct lamellar structure.

In this report, our primary goal was to investigate the tribological behavior of a PTFE/serpentine composite filled with 5 wt. %-30 wt. % of surface-modified serpentine nanoparticles under natural seawater lubrication. To further clarify the tribological mechanisms of the PTFE/serpentine composite, we examined the friction surface topography of the PTFE/serpentine composite and pure PTFE under seawater lubrication. We expect that this study can offer some practical guidance for the application of PTFE-based composites in seawater environments in the bearing field.

II. EXPERIMENTAL PARTS

A. Preparation of Natural Seawater

In this report, natural seawater was obtained from the Qinhuangdao Sea area of the Bohai Gulf with a pH of 7.2 and a salinity of 2.98% measured based on the GB17378.4-2007 standard .

B. Preparation and Modification of the Nanoparticles

Serpentine ore was crushed into fine particles less than 0.5 mm in size using an external force. The nano-serpentine powder is obtained after a certain period of ball-milling using a high-energy ball mill (SamplePrep 8000m, SPEX CertiPreP, USA). Fig. 1 depicts the TEM(JEM-2010, Japan) image of the milled serpentine nanoparticles. The average diameter of the spherical particles is less than 50 nm, and the average column length is less than 150 nm. Fig. 2 and Table 1 illustrate that the EDS (FEI Quanta 200, Holland) of the serpentine nanoparticles primarily consist of three elements, O, Mg, and Si and a small amount of Al and Fe.

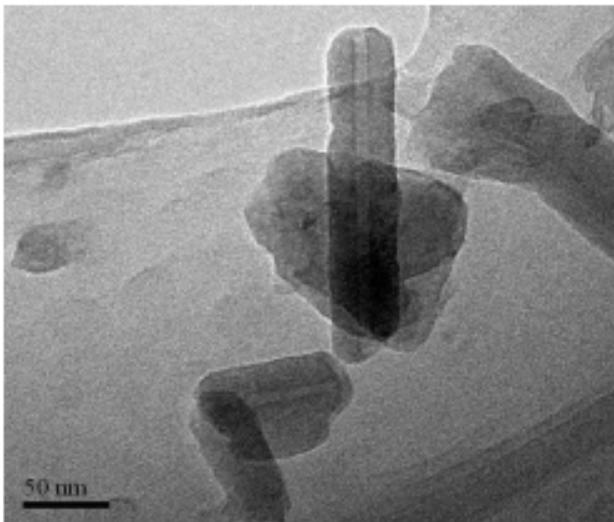


Figure1. TEM image of the nano-serpentine particle

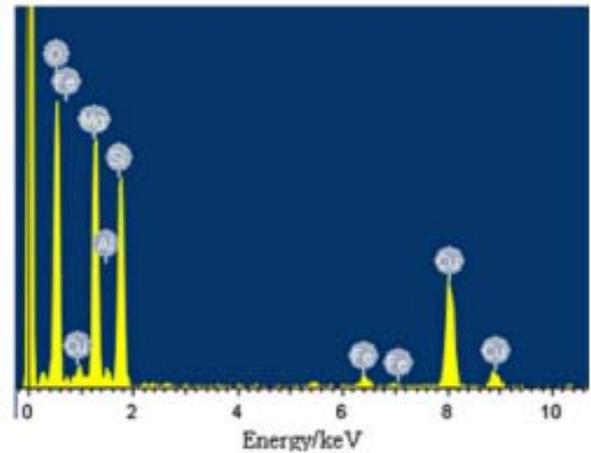


Figure 2. EDS results of the nano-serpentine particle.

TABLE 1: ELEMENT ANALYSIS OF SERPENTINE

Elements	wt.%	at.%
O	46.28	58.77
Mg	27.11	22.66
Al	1.27	0.96
Si	23.32	16.87
Fe	2.02	0.74

The approach modifying the serpentine powder surface proposed in this report is similar to “hydro-metallurgy”, which indicates that the refined serpentine powder and the mixed solution (alcohol, and KH550 whose chemical name is “Amino Functional Silane”) are mixed together for ball-milling. The function of KH550 was to form an isolation layer on the surface of the nano-particles to prevent the agglomeration of serpentine powder. The nano-serpentine powder for the before and after surface modification was characterized using FTIR (E55 + FRA106, Bruker Inc., Karlsruhe, Germany).

C. Preparation of the PTFE/Serpentine Composite

The tensile strength of the PTFE powder (with a particle size of approximately 200 mesh numbers, supplied by Sichuan Chenke Co. Ltd) is greater than 20 MPa, and the elongation is greater than 250 %. After the modified serpentine nanoparticles were filled into the PTFE matrix (with a mass fraction of 5-30 wt %), the mixture was placed into a high-speed mixer and stirred for 5-10 min to ensure a uniform and dispersed mixture. Then, the mixture was compressed in a mold cavity, and a hydraulic pressure was used to form the mixture mold at a constant 50 MPa for 30 min at room temperature. The cold forming sample was sintered in a furnace at a heating rate of 1°C/min, maintained for 90 minutes at 375±5 °C and cooled by the furnace. Lastly, the sample was machined for the next experiment.

The SEM (S-3400n, Hitachi, Japan) images of PTFE/ Serpentine composite surface, reinforced with wt. 25% serpentine nanoparticle, are shown in Fig.3. The Fig. 3a is unmodified composite and Fig. 3b is modified with the

coupling agent KH 550. For the Fig. 3a, the bonding capacity between nano-serpentine particles and PTFE matrix is poor for the unmodified PTFE/Serpentine composite. After the combination of nano-Serpentine and PTFE matrix, it displays “cloud-like” form, which leads to the obvious agglomeration phenomenon of composite. However, for the Fig. 3b, the nano-Serpentine can well dispersed and embedded in the PTFE-based composite, forming a uniform and compact mesh model structure. The network structure can improve the carrying capacity, thereby elevating the comprehensive performance of the composite.

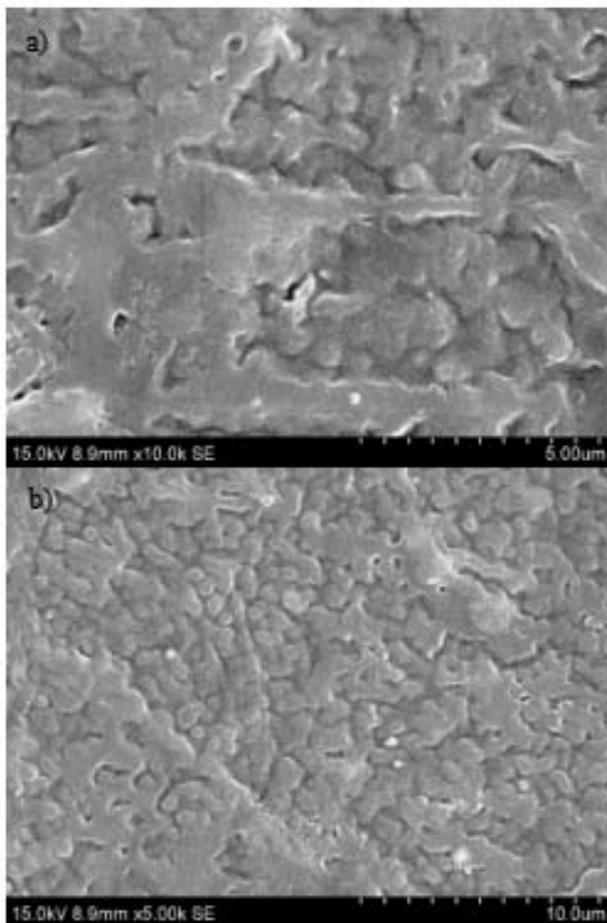


Figure 3. SEM images of the PTFE/Serpentine composite surface, reinforced with wt. 25 % serpentine, a) unmodified and b) modified with coupling agent KH550.

#### D. Water Absorption and Hardness Test of the Composite

The water absorption test was performed based on the ISO62-1999 standard. The PTFE-based composite specimens with different filler contents were immersed into natural seawater for 24 h at room temperature. Then, the weight gain of these samples was measured using an electronic balance with an accuracy of 0.1 mg. The before/after weight gain percent of the samples signifies the water absorption of the PTFE/serpentine composite. The

experimental results are the mean of three measurements.

The hardness was tested based on the ISO7619 standard [26]. The hardness values of these samples were tested before and after seawater immersion for 24 hours. For improving the stability of the data, each experiment was repeated three times and the average used as final experimental result.

#### E. Tribological Tests

The tribological tests were conducted on a MMU-5G (Sida Tester Co. Ltd., Jinan, China) friction and wear tester, as shown in Fig 4a. Fig. 4b illustrates a schematic of the friction pairs. The upper specimen is AISI 1045 steel (composition: 0.42–0.50% C, 0.17–0.37% Si and 0.50–0.80% Mn), which is widely used in various fields, especially in shipbuilding industry. And the lower specimen is the PTFE/serpentine composite. The experimental condition was a sliding velocity of 0.48 m/s under a load of 200 N for a test period of 120 min. Before testing, the PTFE/serpentine composite was burnished by 3000# silicon carbide water sandpaper and the AISI 1045 steel specimen was polished to 0.1–0.2  $\mu\text{m}$  of surface roughness value. Then, it was polished by a polishing machine to create a surface roughness of the sample up of 0.1–0.2  $\mu\text{m}$ . The upper and lower specimens were completely immersed in seawater to simulate seawater lubrication.

In the course of the frictional test, the frictional torque is recorded automatically, and the frictional coefficient is calculated by the friction force. After the test, the sample was cleaned with alcohol and dried in the oven. The wear rate of the PTFE/serpentine composite can be calculated using the formula as follows:

$$\omega = \frac{\Delta m}{\rho PLA} = \frac{\Delta V}{FL}$$

where  $\Delta m$  is the wear loss in g;  $\rho$  is the density of composite in  $\text{g}/\text{mm}^3$ ;  $P$  is the contact pressure in MPa;  $L$  is the sliding distance in mm;  $A$  is the nominal contact area between the upper and the lower specimens in  $\text{mm}^2$ ;  $\Delta V$  is the wear volume; and  $F$  is the experimental contact load. To reduce the dispersion of data, each experiment was repeated three times to obtain the average under the same experimental conditions.

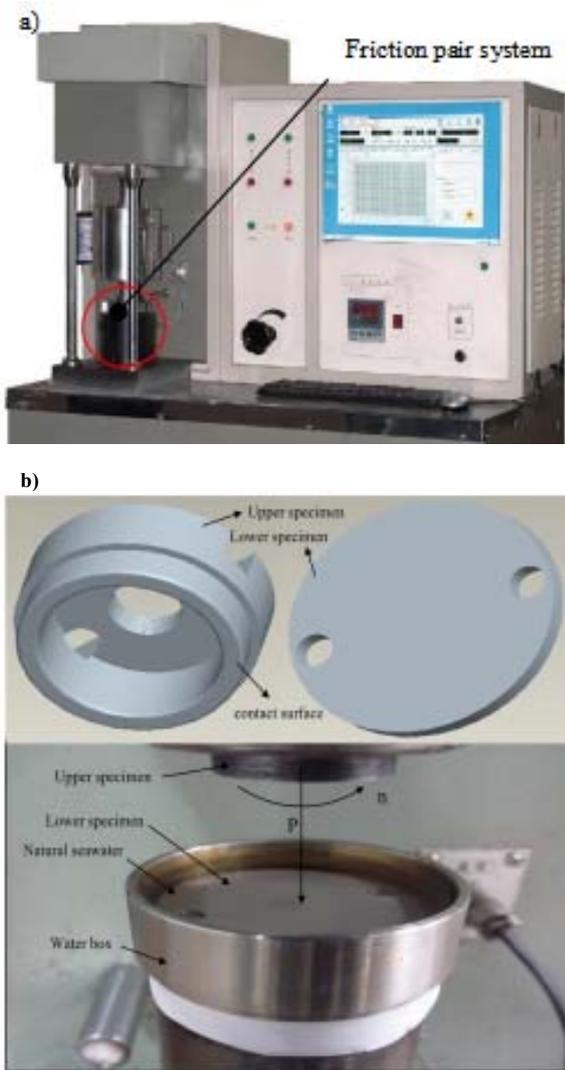


Figure 4. Schematic of the friction pair

*F. Morphology Analysis of the Worn Surfaces*

A scanning electron microscopy and EDS were used to observe the surface morphology of the composite and a AISI 1045 steel ring. To better observe the surface morphology, the surface of the PTFE/serpentine composite was sprayed with gold to make it conductive.

III. RESULTS AND DISCUSSIONS

*A. Infrared Spectrum Analysis*

Fig. 5 depicts the FTIR analysis of the serpentine powders modified using the saline coupling agent KH550. The infrared spectrum of the serpentine powder before modification is observed to be similar to that reported by Central South University [27]. The red curve represents the infrared spectrum of the serpentine powder before modification, while the black one represents the spectrum after modification. A significant difference is found: the position of the vibration peak of the hydroxyl in the modified serpentine surface offsets from 3683  $\text{cm}^{-1}$  to 3670  $\text{cm}^{-1}$ , which is 13  $\text{cm}^{-1}$  higher than that before modification, thus indicating that the polarity of the hydroxyl weakened due to the chemical reaction of KH550 and the serpentine powder. The 3404  $\text{cm}^{-1}$  position of serpentine after the surface modification is the N-H vibration peak and the bending vibration peak of 1596  $\text{cm}^{-1}$  is caused by the serpentine powder with the modified surface. The C-H vibration peak connected with the -CH of KH550 appears at a position of 2922  $\text{cm}^{-1}$  on the surface-modified serpentine due to the effects of KH550. The two absorption bands of the Si-O-Si of serpentine nanoparticles before modification. From the figure, the peak offsets from 1091  $\text{cm}^{-1}$  to 1074  $\text{cm}^{-1}$ , and the peak intensity decreases compared to the infrared spectrum of the serpentine nanoparticles after the surface is modified with KH550. Furthermore, the position of two Si-O bonds of the serpentine after surface modification becomes wider than that of before, which may be due to the new Si-O bond formed by the chemical reaction of KH550 and serpentine. Because the test samples were subjected to repeat washing using anhydrous ethanol and dried at 100 °C, the KH550 adsorbed on the surface of the nano-serpentine particles has already been removed, thus indicating that a chemical reaction occurred on the surface of nano- serpentine particles and formed a new stronger bond, which signifies the success of the surface-modified nano- serpentine.

TABLE 2: PHYSICAL PROPERTIES OF THE PTFE AND THE PTFE/SERPENTINE COMPOSITE IN SEAWATER

Serial number	Before immersed (Shore Hardness)	After immersed (Shore Hardness)	Water adsorption (%)
Neat PTFE	56	56	0.0022
PTFE-5% serpentine	58.5 (+/- 2.35)	58.5 (+/- 2.42)	0.0031
PTFE-10% serpentine	60.5 (+/- 3.91)	60 (+/- 3.25)	0.0044
PTFE-15% serpentine	63 (+/- 3.15)	62.5 (+/- 3.18)	0.0052
PTFE-20% serpentine	65.5 (+/- 2.62)	65 (+/- 2.57)	0.0064
PTFE-25% serpentine	67.5 (+/- 3.36)	67 (+/- 3.34)	0.0070
PTFE-30% serpentine	69 (+/- 2.17)	67.5 (+/- 2.65)	0.0089

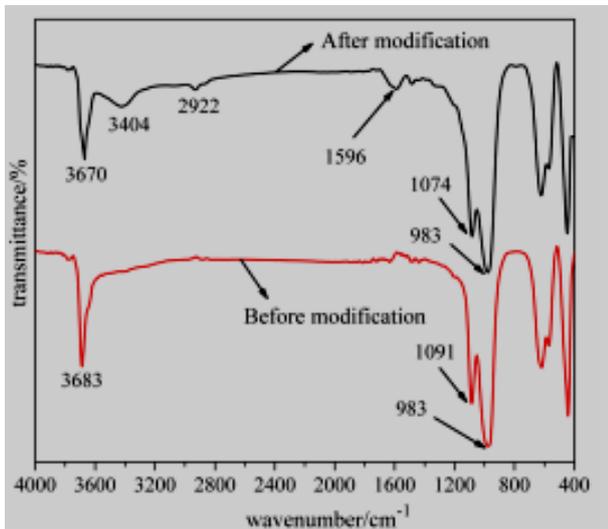


Figure 5. FTIR of the nano-serpentine powder before and after surface modification.

**B. Water Absorption and Shore Hardness**

Table 2 presents the increase in the water absorption rate of the PTFE/serpentine composite when adding surface-modified serpentine. However, the maximum water absorption of the PTFE/serpentine is 0.0089 %. The hardness values of the PTFE filled with surface-modified serpentine is higher than that of the pure PTFE. Before the PTFE/serpentine composite is immersed in seawater, the composite has a maximum hardness value of 69 HD when the filling content of the surface-modified serpentine is 30 %, which is 23.2 % higher than that of the pure PTFE (hardness of 56 HD). This result occurs because of the increasing amount of the surface-modified serpentine, i. e., the "netting" structure of serpentine plays a role in supporting the load. Furthermore, a variation exists in the composite material after being soaked in seawater. Additionally, it can be observed that the hardness value of the PTFE composite is nearly unchanged before and after being immersed in seawater. The water adsorption rate of the PTFE/serpentine composite decreases by two orders of magnitude, compared with that of the bearing material, i. e., nylon (PA6) [28]. Thus, the plasticizing effect of these composites caused by water absorption can be negligible, and it can be concluded that the plasticizing effect is not a key factor in influencing the tribological properties of the PTFE/serpentine composite under seawater lubrication conditions.

**C. Friction and Wear Properties of the Composite**

Fig. 6 illustrates the changes in the friction coefficient curve of the PTFE/serpentine composite filled with varying amount of surface-modified serpentine under seawater lubrication. The figure indicates that the friction coefficient

of pure PTFE is 0.039. The friction coefficient of the PTFE/serpentine composite firstly decreases and then increases with an increase in the surface-modified serpentine content. The lowest friction coefficient value is 0.009 when the amount of serpentine is 20 %, which is 77 % lower than that of the pure PTFE, thus indicating optimal anti-friction characteristics. Fig. 7 illustrates the changes in the curve of the wear rate of the PTFE/serpentine composite in seawater lubrication. From the figure, the wear rate of the PTFE composite gradually decreased, when the amount of surface-modified serpentine was less than 15 % and increased when the amount was more than 15 %. The lowest wear rate of the composite is  $7 \times 10^{-6} \text{ mm}^3 \cdot (\text{N} \cdot \text{m})^{-1}$  with 15 % serpentine, and the wear resistance is an order of magnitude larger than that of the pure PTFE ( $8.1 \times 10^{-5} \text{ mm}^3 \cdot (\text{N} \cdot \text{m})^{-1}$ ). Furthermore, the wear rate of the PTFE/serpentine composite, compared with that of the pure PTFE, greatly decreases and the wear resistance increases nearly 12 times under the same condition.

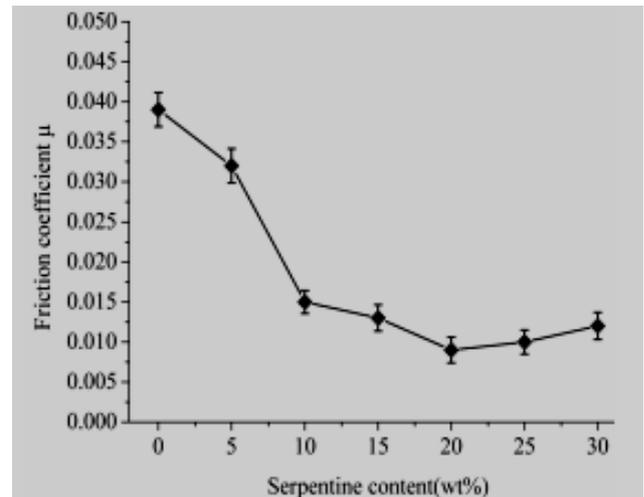


Figure 6. Friction coefficient of the PTFE/serpentine composite.

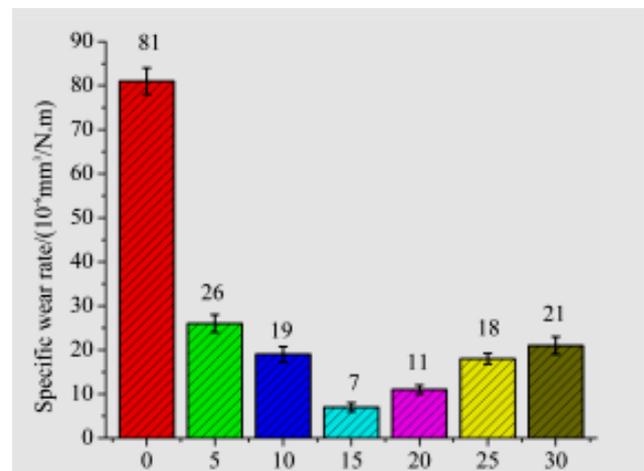


Figure 7. Wear rate of the PTFE/serpentine composite.

*C. Analysis of Friction and Wear Mechanism*

The worn surfaces of the PTFE and composite under seawater lubrication are illustrated in Fig. 8. From the figure, a wide furrow and a large number of cracks are generated on the worn surface of the pure PTFE (Fig. 8A), which is similar to an earlier study [11]. The worn surface of the PTFE had considerable fatigue wear because of the alternating cyclic stress, thus resulting in a "shell-like" crack. When the amount of the surface-modified serpentine was 5 % (Fig. 8B), different shades of furrows, slight plastic deformation and certain degree of abrasive wear were observed on the worn surface of the composite. Fig. 8C depicts the worn surface morphology of the PTFE-based composite with 15 % of the surface-modified serpentine. It is evident that the worn surface is relatively smooth and the furrows on the surfaces are small and shallow. Fig. 8D depicts the worn surface morphology of the composite with 30 % of the surface-modified serpentine.

*D. Analysis of the Worn Surface*

The SEM morphology of the worn surface for both the pure PTFE and the PTFE composites sliding against AISI 1045 steel is depicted in Fig. 9. Different shades of furrows are generated on the steel ring surface along the sliding direction (Fig. 9a). This is the result of the corrosive effects of seawater, which increase the surface roughness of the steel ring, thus resulting in a relatively high friction coefficient and wear rate for the pure PTFE in seawater. The furrows effect on the steel ring surface decreased when the amount of surface-modified serpentine was 5 % (Fig. 9b). A layer of white transparent corrosion generated on the steel ring surface. Hence, the corresponding wear rate of the composites decreases compared with that of the pure PTFE. When the amount of the surface-modified serpentine was 15 % (Fig. 9c), the wear surface was smooth and clean. Additionally, its surface roughness was the lowest, which resulted in the lowest wear rate of the composite. When the filling content of the surface-modified serpentine is 30% (Fig. 9d), the steel ring surface is covered by a layer of transparent material. Furthermore, the furrows generated on the steel ring surface become wide, shallow and non-directional.

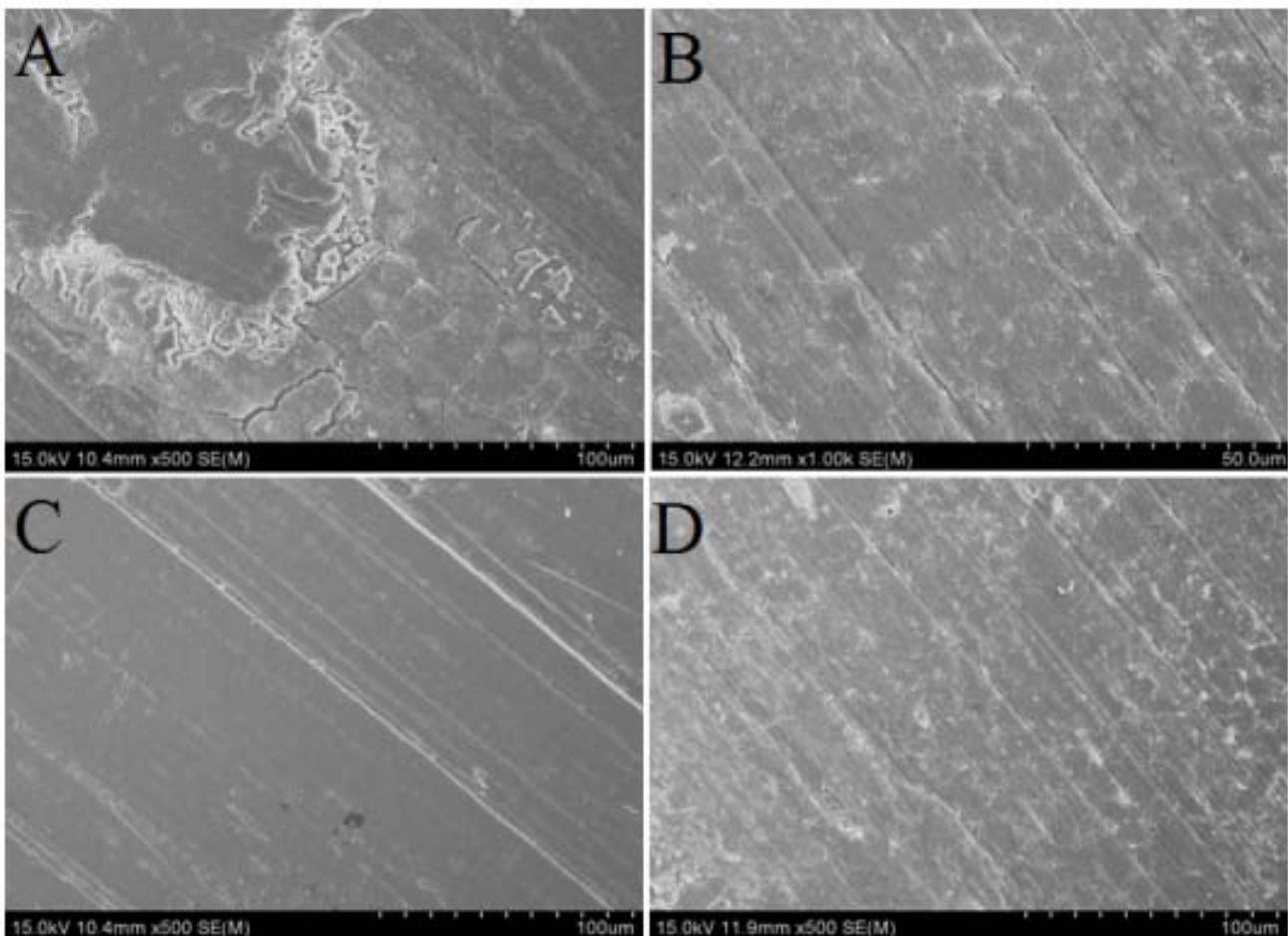


Figure 8. SEM images of the worn surface of the composites after the friction tests under seawater lubrication

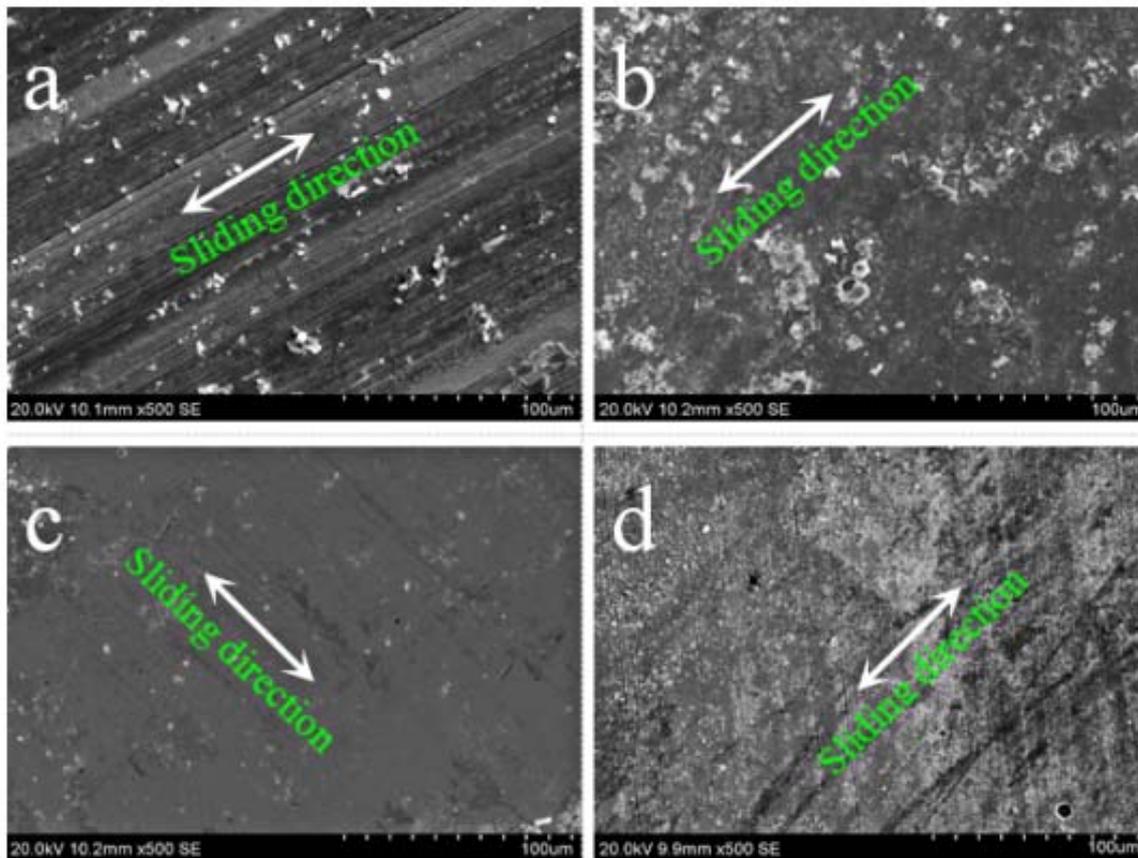
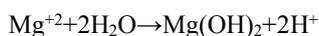
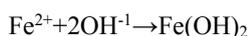
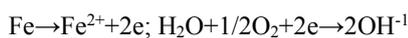


Figure 9. SEM micrographs of the worn surface of AISI 1045 steel after friction tests under seawater lubrication.

Fig. 10 presents the EDS analysis of the steel ring surface. From the figures, the elements on the surface of the steel ring mainly consist of C, O, and Fe, and the characteristic element F of the PTFE is not observed. This phenomenon proves that under seawater lubrication conditions, it is difficult for the PTFE to form a self-lubricating transfer film on the AISI 1045 steel substrate surface. In other words, in this case, the seawater lubrication film is the most effective. Here, the serpentine nanoparticles primarily play a role in increasing the bearing capacity of the composites and changing the contact state of the worn surface. Additionally, the EDS analysis of the steel ring surface indicates that NaCl and/or Mg(OH)<sub>2</sub> is deposited on the surface, namely the white transparent material mentioned above [29]. These chemical reactions can be described by the equation as follows:



However, not all of the composite materials are similar. As depicted in Fig. 10a, b and d, magnesium was not observed on the surface of the steel ring. When the filling content is

15%, six types of elements, C, O, Fe, Na, Mg and Cl, were detected on the wear surface (Fig. 10c). The divalent Mg<sup>2+</sup> from the serpentine particles can produce Mg(OH)<sub>2</sub> due to the friction chemical reaction during the friction process. The reaction can isolate the friction pair, thus inhibiting the corrosion effects of the steel ring. The interaction of the chemical reaction and the seawater lubrication film result in a significant decrease in the wear rate of the composites.

The effects of the serpentine nanoparticles and the seawater corrosion cannot be ignored, which will help us to further understand the mechanism of friction and wear. As previously mentioned, because of the corrosive properties of seawater, it is difficult to form a transfer film. Thus, pure PTFE indicates a high wear rate. Meanwhile, the serpentine nanoparticles added into the PTFE that break from the contact surface can also play a role of "micro-bearing", which is the reason for the macroscopically lower wear rate of the composite [30, 31]. However, the increase in the filling amount indicates that the number of nanoparticles breaking off from the contact surface increases, which would inevitably cause sliding behavior. The random orientation of the scratches clearly demonstrates this performance, as indicated in Fig. 8d. Therefore, a relative

higher wear rate was examined. At the same time, this

effect may also destroy the formation of the transfer film.

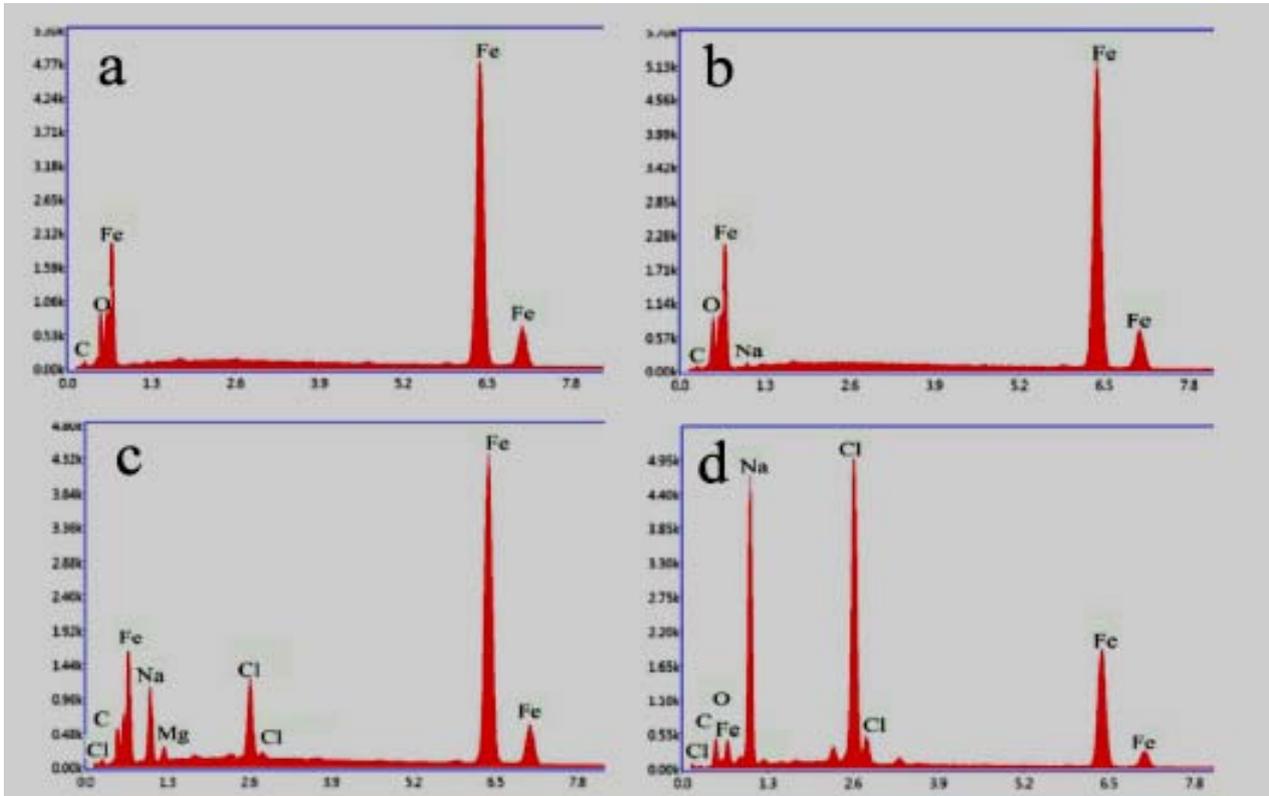


Figure 10. EDS analysis of the worn surface of AISI 1045 steel after friction test.

#### IV. CONCLUSIONS

The techniques of TEM, SEM, EDS, and FTIR combined with a series of tribological tests were used to study the tribological properties of PTFE-based composites filled with surface-modified nano-serpentine under natural seawater lubrication. The following conclusions can be drawn.

The water absorption rate of PTFE and PTFE-based composites filled with surface-modified nano-serpentine is extremely low in seawater, and the hardness of the composites hardly changes before/after seawater immersion. Therefore, water absorption and plasticization are not the primary factors that influence the tribological behaviors of composites under natural seawater.

The incorporation of the surface-modified serpentine remarkably improves the wear resistance of the PTFE. When the filling content of is 15 %, the lowest wear rate of the PTFE/serpentine composite can be obtained. Due to the corrosive properties of seawater, the transfer film was not observed on the steel surface of the ring. Moreover, the filling content of the nano-serpentine and the friction chemical reactions are the primary mechanisms of friction and wear.

#### ACKNOWLEDGEMENT

The authors acknowledge the financial support of the Open Foundation of Key Laboratory for Aerospace Science and Technology and the Natural Science Foundation of Hebei Province (Grant No. E2016411005). Furthermore, special thanks are given to the editors and the reviewers for their valuable instructive comments and suggestions.

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