Effect of Square Texture on Tribological Properties of Nano-SiO$_2$/POB-PTFE Composites

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Abstract. In order to study the design problem of piston rings in Stirling engine, on the basis of filling PTFE with Nano-SiO$_2$ and POB and preparing the GCr15 contact surface with square texture by HM20-I laser marking machine, experiments were carried out on LSR-2M wear tester by indirect weighing and in-situ observation methods. Optical microscope (OM) and scanning electron microscope (SEM) were used to observe the evolution of Nano-SiO$_2$/POB-PTFE composites' transfer film on contact surface. The results showed that the square texture would shorten the running-in and transitional periods of the composites' tribological process, accelerate into the stationary period. The formation process of the composites' transfer film on the square textured contact surface was also different from smooth contact surface. Although the square texture would increase wear rate, its ability to store wear debris is more conducive to the formation of reliable, uniform and continuous transfer film with a same friction direction. Obviously, reasonable design of surface texture can effectively improve the wear resistance of sealing parts made of filling modified PTFE composites, thus providing theoretical guidance for the seal design of Stirling engine piston ring.

1. Introduction

The Stirling engine is the core component of the dish-type solar thermal power generation system. It uses gas (hydrogen or helium) as the working medium and works in a closed regenerative cycle, so the reliability of the seal is essential. However, the working environment of the piston ring is high-speed, high-pressure, high-temperature and oil-free, which is easy to wear and deteriorate sealing performance or even sealing failure. Therefore, the seal design of Stirling engines piston rings is critical.

Polytetrafluoroethylene (PTFE) is widely used in seals for its excellent chemical stability and self-lubricating properties. However, it has disadvantages such as poor wear resistance, low load-bearing capacity, and easy creep[1, 2]. In order to increase the service life of PTFE, researchers have done much research on filler modification. The study found that hard filler Nano-SiO$_2$ is easy to mechanically interlock with the contact surface during the friction process and promotes transfer film’s formation [3]. Soft filler POB can reduce the strength and hardness of composites, and effectively prevent fracture of the PTFE ribbon structure during friction process[4]. It also has strong adhesion. Therefore, the synergistic anti-friction effect of Nano-SiO$_2$ and POB can promote the formation and adhesion stability of transfer film [5], thereby improving the wear resistance of the PTFE composites.

In terms of friction pair design, researchers have studied the influence of surface engineering on the tribological properties of PTFE composites, mainly including working conditions[6], surface texture[7, 8, 9], surface roughness[10, 11] and surface coating[12]. Chen [13] found that the square micro-groove texture has the lowest friction under lubricating conditions than the hexagonal and...
triangular micro-groove textures. It is because the $\perp$ and $\parallel$ directional grooves have the best effect on friction. Slawomir [14] used pin-disc friction and wear tester to conduct a tribological experiment of one-way sliding under non-lubricated conditions and found that the surface texture reduced the friction coefficient by 2.2 times. Li [15] used laser processing method to prepare grid, ring, and star micro-textures on the CuSn6 alloy surface. Study found that these three textured surfaces effectively reduced the friction coefficient of PTFE and increased the wear rate before polishing. In addition, study have also found that a reasonable texture design has positive effect on reducing the friction and wear of the friction pair[16, 17, 18], which including the size, shape, depth, and surface position of the texture and other parameters[19], so it has a theoretical guiding role for actual engineering processing. In summary, researchers generally use friction coefficient and wear rate as indicators to evaluate the tribological performance of friction pairs with different surface textures. However, they seldom study the evolution process of transfer film on the contact surface during tribological process.

In this paper, for the friction pair composed of Nano-SiO$_2$/POB-PTFE composites and GCr15, an experimental study was conducted by indirect weighing and in-situ observation methods on LSR-2M tester. The purpose is to explore the influence of square texture on the transfer film evolutionary process and friction and wear behavior of the filling modified composites, further to provide a theoretical basis for the seal design of Stirling engine piston ring.

2. Experimental Part

2.1. Raw Materials

The brand of PTFE suspension powder is M18F, which was produced by Japan's Daikin Co., Ltd. Its average particle size is 25 $\mu$m, bulk density is 0.33 g/cm$^3$, and apparent density is about 0.47 g/mL. Polyphenylester (POB) has an average particle size of 15 $\mu$m, produced by Zhonglan Chenguang Chemical Research Institute Engineering Plastics Company. Nano-SiO$_2$ powder has an average particle size of 20 nm and was produced by Nanjing Xianfeng Nano Material Technology Co., Ltd.

2.2. Sample Preparation

PTFE, POB and Nano-SiO$_2$ were formulated according to Table 1. High-speed mechanical mixer was used to mix them 5 times, and 45-mesh sieve was used to remove particles due to mutual attraction of nanoparticles, followed by molding after 24 h of resting. The blank size was 8 mm×30 mm, and the pressing pressure was 30 MPa for 3 min. At the end of removing the pressure, the sample edges and molds were removed. After 24 h of resting, the sintering temperature was 374 °C, the heating rate was 2 °C/min, and the holding time was 240 min. Sintering later, the sample was naturally cooled to room temperature with the furnace. Finally, the blank was machined to 6 mm×10 mm, sonicated with acetone for 15 min, and dried.

Table 1. Sample proportion(wt.%).

| PTFE(wt.%) | POB(wt.%) | Nano-SiO$_2$(wt.%) |
|-----------|-----------|-------------------|
| 84        | 15        | 1                 |
2.3. Preparation of the Contact Surface

2.3.1. Preparation of smooth contact surface

![Image of smooth contact surface](image1)

Figure 1. Schematic diagram of the smooth contact surface.

Under the light microscope, there was no trace of processing on the mirror smooth contact surface. In order to better observe the transfer film and analyze its evolution process, the contact surface was processed into mirror smoothness in this study. A grinding method prepared a mirror-smooth surface on the surface of a 50 mm×6 mm GCr15 bearing steel plate. The surface roughness was 0.004-0.006 μm.

2.3.2. Preparation of textured contact surface

The HM20-I laser marking machine was used to prepare square texture on the surface of a GCr15 plate with the size 50 mm×6 mm. The square's side length was 200 μm, the depth was 10 μm, and the vertical and horizontal spacing was 400 μm. After the texture was prepared, the MicroXAM-800 three-dimensional surface profiler was used to characterize the two-dimensional morphology and contour curve, as shown in Figure 2.

![Image of texture morphology and contour curve](image2)

Figure 2. Texture morphology and contour curve (a) Two-dimensional topography; (b) Contour.

2.4. Friction and Wear Test

The schematic diagram of the friction pair is shown in Figure 3, which the bolt's size (sample) was 6 mm×10 mm. The environmental condition of the test was room temperature, the load was 170 N, the reciprocating speed was 0.033 m/s and the reciprocating stroke was 10 mm. The test was periodically interrupted, and the sample was taken out after the interruption. It was weighed on a balance with an accuracy of 0.01 mg. Using an optical microscope, the center position of the wear scar on the contact surface was observed to track the morphological evolution of the transfer film. The interruption time increased with the progress of the experiment. The intermittent periods of the two experiments were kept the same for comparison.
3. Results and Discussion

3.1. Tribological Performance Analysis

The wear volume of the Nano-SiO$_2$/POB-PTFE sample on the smooth and square textured contact surfaces are shown in Figures 4 (a) and (b). It can be seen from the figures that the friction and wear process of the contact surface can be divided into three stages. The first stage is the running-in period, the wear rate decreases monotonously with the increase of sliding distance. The second stage is the transitionary period with almost zero wear. The third stage is the stationary period, and the wear rate of the composites is almost unchanged[20]. The wear rate of the Nano-SiO$_2$/POB-PTFE sample on the square textured contact surface is shown in Figure 5. The stationary period of the friction and wear process can be observed more clearly.
Figure 5. Wear rate of on the square texture surface.

The wear volume and average wear rate of the Nano-SiO$_2$/POB-PTFE sample on the smooth and square textured contact surfaces are shown in Figures 6 (a) and (b). It can be seen from Figure (a) that the square texture slightly shortens the running-in and transitionary periods than the smooth contact surface, and speeds up into the stationary period. The Nano-SiO$_2$/POB-PTFE sample wears more when tribological process of square texture enters the stationary period. After the test, the total wear volume of the sample is about 5 times that on the smooth contact surface. As shown in Figure (b), the smooth contact surface has a lower average wear rate than the square texture compared with the whole test period.

Figure 6. Wear volume and average wear rate on the smooth and square textured contact surfaces

The friction coefficient and average friction coefficient of the Nano-SiO$_2$/POB-PTFE sample under the smooth and square textured contact surfaces are shown in Figures 7 (a) and (b). It can be seen from Figure (a) that the friction coefficient on the smooth surface fluctuates violently and frequently during 0-750 minutes. Then, with the transfer film’s appearance, the friction coefficient tends to be stable and shows a downward trend. However, the friction coefficient of the square texture has been relatively stable and shows an upward trend. Due to the appearance of the transfer film, it also has not decreased and is generally between 0.16-0.22. When the experiment was conducted for 1500min, the friction coefficient of the smooth contact surface continued to decrease until it was lower the square texture. As shown in Figure (b), compared to the entire test period, the average friction coefficient of the square texture is lower than that of the smooth contact surface.
The friction and wear test results show that the square texture stabilizes the friction coefficient of the friction pair, reduces the average friction coefficient, but increases the wear rate. The wear rate’s increase is due to stress concentration at the edges of the textured grooves and even cutting action [21]. The instantaneous stress may cause severe friction and wear when the sample moves from the textured pit to the flat area.

3.2. Analysis of the Evolution Process of the Transfer Film

3.2.1. Running-in period

The surface topography of the Nano-SiO$_2$/POB-PTFE sample after sliding 9.9 m on the smooth and square textured contact surfaces are shown in Figures 8 (a) and (b). The wear rate during this process is $4.7 \times 10^{-5}$ mm$^3$/N$\times$m and $6.47 \times 10^{-5}$ mm$^3$/N$\times$m.

As shown in Figure (a), the morphology of the transfer film on the smooth contact surface is mainly composed of linear wear debris, and there is a tendency to turn from linear to block. It is generally believed that the flake wears debris acts on the pressure between the grinding surfaces. The bottom wear debris is rolled and ironed, and its wear mechanism is mainly adhesive wear [12]. After several subsequent tests, the linear transfer film appeared to a slightly peeling off, but most of the composites still adhered firmly to the contact surface.

As shown in Figure (b), no more wear debris are observed in the plane area of the square textured contact surface, but some larger pieces of wear debris adhere to the contact surface. This is because textured pits store wear debris, resulting in a less obvious distribution of wear debris on the plane area. However, as the test progresses, when the amount of wear debris stored in the pit reaches a certain amount, the wear debris will overflow to form a thin strip-shaped transfer film, and parts adhere to the flat area of the contact surface.
3.2.2. Transitionary period

For the Nano-SiO₂/POB-PTFE sample during the transitionary period, the representative pictures of the transfer film surface morphology on the smooth and square textured contact surfaces are shown in Figures 9 (a) and (b).

As shown in Figure (a), the transfer film morphology on the smooth contact surface undergoes a process of changing from a linear morphology to a block morphology. Tiny brownish-yellow block wear debris adhere to the transfer film and metal counterpart surface. There is a tendency to form wide stripe-shaped transfer films.

As shown in Figure (b), the transfer film comprises small wear debris and larger pieces of wear debris in the upper left area. The area formed by the transfer film is mainly located in the flat area of the square textured contact surface, parallels to the rubbing direction. As the test continues, the transfer film will be more pronounced, its area will be further expanded, and the sample will enter a steady-state cycle.

3.2.3. Stationary period

For the Nano-SiO₂/POB-PTFE sample during the stationary period, the representative morphology pictures of transfer film surface on the smooth and square textured contact surfaces are shown in Figures 10 (a) and (b).

As shown in Figure (a), brown-yellow broad stripe transfer films are formed with the block transfer film on the smooth surface. The transfer film is uniformly and continuously distributed, resulting in a low wear rate in a particular process. The contact surface has good reflectivity, and the transfer film still has the phenomenon of shedding and growing in the subsequent tests.

As shown in Figure(b), with the experiment progresses, block-like and broad-striped transfer films are formed on the square textured contact surface. The commonality of two kinds of contact surface is
that the transfer film formed by the wear debris with good adhesion can be retained for a long time. Then the friction pair is converted from the friction between "metal-polymer" to "polymer-polymer", thereby effectively improving the friction and wear performance. In addition, block-like and broad-stripe transfer films are observed during this period. The difference is that the smooth contact surface has good reflective performance as the test progresses, the square texture is relatively poor. But the transfer film on square texture has high coverage, uniform and continuous distribution, basically covering the area observed by the light microscope.

![Figure 10](image1.png)

**Figure 10.** Representative morphology images of contact surfaces during the stationary period

### 3.3. Transfer Film Analysis

After the test, the SEM image of the transfer film morphology at the center area of the wear scar on the smooth and square textured contact surfaces are shown in Figures 11 (a) and (b). The vertical direction in the figure is the reciprocating sliding direction of the sample. According to Figure (a), a large number of mottled transfer films can be observed. Their distribution is relatively uniform, the adhesion on the contact surface is good, and the transfer film tends to aggregate to form broad stripes. It can be seen from Figure (b) that only part of the transfer film adheres to the contact surface, and its distribution is very uniform.

The scanned images of the F element distribution on the smooth and square textured contact surfaces are shown in Figures 12 (a) and (b). According to Figure (a), it can be observed that a large number of F elements of the composites wear debris are evenly distributed on the smooth surface. This indicates that the wear debris adheres well to the mating surface. It can be seen from Figure (b) that a large number of F elements of the composites wear debris are stored in the textured pits, and evenly distributed in the planar area of the textured surface. This indicates that a large amount of wear debris is stored in the square texture, a small amount is distributed in the flat area.

![Figure 11](image2.png)

**Figure 11.** SEM images of contact surfaces
After the test, the three-dimensional topography of the transfer film on the smooth and textured contact surfaces are shown in Figures 13 (a) and (b). The photographed area is 6 mm×1.16 mm in the middle of the wear scar. As shown in Figure (a), the transfer film is evenly distributed, and the overall coverage is well. However, there are more peaks, and the peak-to-valley drop is more prominent than in Figure (b). As shown in Figure (b), the evenly distributed peaks are about 13 μm, most of which are clustered near the square texture. The peaks are continuous and not much different, which indicates that uniform transfer film is produced.

4. Conclusion
1) The square texture will shorten the running-in and transitional periods of the Nano-SiO₂/POB-PTFE composites, accelerate into the stationary period. It also promotes the formation of transfer film and significantly improves the wear resistance of the composites.

2) The square texture results in a different transfer film formation process from the smooth contact surface. During the running-in period, the square texture stores wear debris until overflows. During the transitional period, the transfer film begins to form and mainly adheres to flat part of the textured surface. During the stationary state, the wear rate is low and stable, and the block and wide stripe transfer films are formed on the contact surface.

3) Although the square texture will increase the wear rate, its grooves can store wear debris, therefore the nanoparticles of wear debris can form mechanical interlocking force with the groove’s bottom and rough side peaks. It can reduce the amount of wear debris on friction interface, avoid the plowing effect of hard particles on the transfer film. Moreover, it is more conducive to the formation of reliable, uniform and continuous transfer film with a same friction direction.

4) In engineering practice, the appropriate surface texture design can significantly improve the tribological performance. Therefore, the next step is to optimize the textured geometric parameters to provide theoretical guidance for the seal design of Stirling engine piston ring.
5. References

[1] Mazza L, Trivella A, Grassi R and Malucelli G 2015 *Tribology International* A comparison of the relative friction and wear responses of PTFE and PTFE-based composite when tested using three different types of sliding wear machines p15-21.

[2] L. Huang and Z. B. Sun 2000 *Acta Material Composite Sinica* Study on the frictional and mechanical performance of PTFE composites 17 p54-57.

[3] Burris D L, S. Zhao, Duncan R, Lowitz J and Sawyer W G 2009 *Wear* A route to wear-resistant PTFE via trace loadings of functionalized nanofillers 267 p653-660.

[4] G. Xie, L. F. Cai and C. F. Huang 2007 *Material Development and Application* Research on the mechanical and tribological properties of polyphenylene ester filled polytetrafluoroethylene composites 22 p27-37.

[5] D. Y. Yang, X. W. Xiong and G. Gao 2021 *Lanzhou University of Technology* Research on the growth process of the friction transfer film of Nano-SiO$_2$/POB modified PTFE composites.

[6] B. Y. Song, L. Gu and E. H. Xing 2004 *Journal of Harbin Institute of Technology* Friction and wear properties of GCr15 steel under vacuum condition 36 p239.

[7] G. Gao, J. Gong, R. H. Li, H. G. Wang, J. F. Ren and S. S. Chen 2020 *Chinese Journal of Tribology* The effect of surface texture on the friction and wear behavior of PTFE composites 40 p697.

[8] K. F. Song 2020 *Yanshan University* Fabrication of complex-textured PTFE surface by compression and sintering and its tribological properties.

[9] H. Y. Zhang 2018 *Hefei University of Technology* The effect of metal surface texture on the wear of PTFE-Alumina composites.

[10] G. Gao, R. H. Li, J Gong, H. G. Wang, D. Y. Yang, J. F. Ren and S. S. Chen 2020 *Journal of Sichuan University (Engineering Science Edition)* The effect of contact surface roughness on the tribological properties of Nano-SiO$_2$ modified PTFE composites 52 p207.

[11] S. J. Ye, Q. Fan, L. Y. Deng and Y. H. Wang 2010 *Lubrication & Sealing* The effect of copper powder on the mechanical and tribological properties of PTFE composites 35 p64.

[12] Soltani-Kordshuli F, Okyere D, J. CHEN, Miller C, Zou M 2021 *Surface and Coatings Technology* Tribological behavior of the PDA/PTFE + Cu-SiO$_2$ nanoparticle thin coatings 409 126852.

[13] P. Chen, J. L. Li and Y. L. Li 2018 *Central South University* Effect of geometric microgroove texture patterns on the tribological performance of stainless steel 25 p331-341.

[14] Slawomir W, Waldemar K, Pawel P, Drabik J and Rogos E 2017 *Tribology International* Effects of surface texturing and kind of lubricant on the coefficient of friction at ambient and elevated temperatures 117 p174-179.

[15] J. Li, S. Liu, A. Yu and S. Xiang 2018 *Tribology International* Effect of laser surface texture on CuSn6 bronze sliding against PTFE material under dry friction 118 p37-45.

[16] C. T. Zhang, M. L. Wang and X. L. Wang 2017 *Surface Technology* The effect of surface texture on the wear behavior and mechanism of metal-polyoxymethylene friction pairs 46 p9-14.

[17] R. F. Yu and W. Chen 2017 *Chinese Journal of Mechanical Engineering* Research status and prospects of surface texture in the field of industrial tribology 53 p100-109.

[18] L. L. Wang, W. Zhang, X. T. Zhao, Z. K. Liu and M. X. He 2020 *Friction Journal of Chinese Academy of Sciences* The effect of micro-texture size on the friction and wear properties of bearings.

[19] W. A. Wang, Z. Q. Liu, D. L. Chen, Z. M. Xie, and J. L. Song 2021 *Advances in Materials Science and Engineering* Influence of different surface texture parameters on the contact performance of piston ring-sleeve friction pair of hydraulic cylinders.

[20] J. Ye, B. Tao, W. Sun, Haidar D R, Alam K I, K. Liu and Burris D L 2018 *Tribology Letters* The competing effects of counterface peaks and valleys on the wear and transfer of ultra-low wear Alumina–PTFE 66 p12.

[21] S. Zhao and X. L. Wang 2015 *Tribology* The effects of surface texture on the wear properties of mechanical seals made of metal and polymers 35 p761-767.